BAW 1014

LIQUID METAL FUEL REACTOR EXPERIMENT

RESEARCH AND DEVELOPMENT PROGRAM

JUNE 1957

THE BABCOCK & WILCOX COMPANY

ATOMIC ENERGY DIVISION

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UNITED STATES ATOMIC ENERGY COMMISSION
BY
THE BABCOCK AND WILCOX COMPANY

RESEARCH AND DEVELOPMENT PROGRAM

FOR LMFRE

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I. INTRODUCTION

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The properties of the U-Bi fuel system have been under investigation at Brookhaven National Laboratory since 1950. These studies, for the most part, represent exploratory research aimed at:

1) developing basic knowledge on the stability and corrosiveness of U-Bi fuel combinations in contact with various reactor construction materials; and

2) proving the feasibility of selectively removing many of the fission products from U-Bi fuel by volatilization and salt extraction.

With respect to the metallurgical phase of the Brookhaven program, considerable information has been developed on the uranium-graphite reaction, effects of the principal constituents in materials of construction on the solubility of uranium in bismuth, corrosion and mass transfer by liquid bismuth and the mechanism of corrosion inhibition.

The feasibility of the technique of removing volatile fission products by degassing the circulating fuel has been demonstrated by operations of an experimental test loop in the Brookhaven reactor. Other tests along the same lines have indicated that some of the non-volatile elements can be removed selectively by fused halide salt extraction procedures and a few tests have given evidence that these elements, including uranium, can be reduced from the fused-salt phase back into the liquid metal stream.

In many instances, the information obtained at Brookhaven is general in character and lends itself only to qualitative appraisal. Consequently, at this stage of the investigations the Brookhaven National Laboratory experimental data create an expectation of favorable behavior, but these data do not reasonably insure it for any specific set of design parameters considered for the Liquid Metal Fuel Reactor Experiment.

Because of these factors, The Babcock & Wilcox Company considers it necessary to carry out the following Research and Development Program, the purpose of which is to provide specific engineering information required for the design, construction and operation of the Liquid Metal Fuel Reactor Experiment.

II. MATERIALS TESTING

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A. Introduction

The principal objective of this program is to confirm the suitability of Croloy 2 1/4 (2 1/4 chromium-1 molybdenum steel) and graphite as construction materials for the Liquid Metal Fuel Reactor Experiment under defined conditions of temperature, temperature gradient, velocity, chemical environment and radiation, and to investigate Croloy 1 1/4 (1 1/4 chromium-1/2 molybdenum steel) as a possible alternate material. Independent and combined effects of these factors will be determined by this program primarily based on forced circulation liquid bismuth corrosion test loops. Testing will be done under two separate conditions with respect to maximum temperature and temperature gradient and will include parallel programs for the Croloy 2 1/4 and Croloy 1 1/4 steels.

Under reference temperature conditions 22 loops will be operated from eight loop stations at a maximum temperature of 885 F with a temperature gradient of 135 F. Eleven of these loops will be built of Croloy 2 1/4 and eleven of Croloy 1 1/4.

A loop station will include instrumentation, pumps, heaters, and melt, sampling, and sump tanks. Operation of more than one loop from a single station will be accomplished by sequential runs, replacing only the integral loop components after the completion of each previous run.

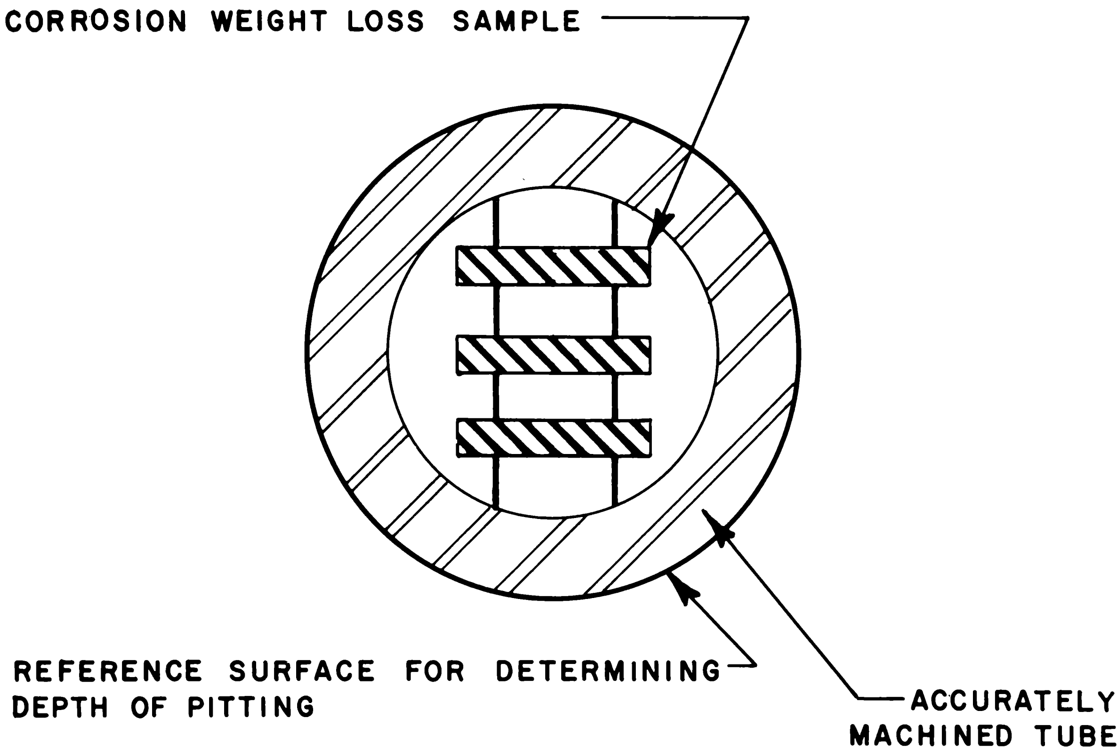
In this manner, two of the eight stations will be utilized for six 1500 hour loop tests each, to determine the effects of heat treatment, velocity, and additives to the liquid bismuth on the corrosion resistance of these steels. Two more stations will be required for corrosion rate determination accomplished by operating three loops for test duration of 1500 hours, 3000 hours, and 5500 hours at each station. Four loops, to be run without interruption for 10,000 hours will occupy the four remaining stations. These will be essentially demonstration runs for proof testing the reference conditions.

Identical testing will be performed under alternate temperature conditions at a maximum temperature of 975 F with a temperature gradient of 225 F.

All loops will contain samples of graphite, weldments, and loop material in hot and cold sections. These samples are of the type shown in Figure 1 and will permit quantitative determination of the extent of attack.

To provide preliminary information which can be applied to the forced circulation loop testing, a program of dynamic and static tests, termed supporting tests, will be carried out. The information and data obtained from these tests will serve in modification or corroboration of the proposed forced circulation loop test conditions and will include investigation of the effects of heat treatment, additives, corrosion products, and fission products on the corrosion resistance of Croloy 2 1/4 and Croloy 1 1/4 in liquid bismuth.

FIG. I : CORROSION TEST SAMPLE SKETCH



Apart from the corrosion test loops, a forced circulation utility loop will be constructed. Proof testing graphite to graphite joints, graphite to metal joints, pumps, valves, and bearings will be performed with this loop.

Additional supporting static and dynamic tests will be run to evaluate the suitability of other materials of construction, to investigate certain basic problems related to liquid bismuth corrosion, and to obtain information further advancing the materials technology of the LMFR concept. These alternate supporting tests will include: investigation of materials for control rods, port thimbles, and chemical reprocessing equipment; studies of ZrN protective film formation, stability and effectiveness, and other factors influencing mass transfer; determination of various properties of graphite. Where applicable, the results obtained of proof testing from the alternate supporting test will be carried out with the utility loop.

The ultimate test of the materials of construction will be the determination of their performance under irradiation. Due to the scarcity of in-pile testing facilities, radiation data will be obtained from a minimum of testing. Contingent upon accessibility to reactor test space of adequate flux level, three in-pile forced-circulation uranium-bismuth loops will be built and operated for periods of about 5,000 hours. From these loops, will be determined the effects of radiation on mechanical, physical and corrosion properties of the material of construction and the performance of pumps, valves, bearings, and expansion joints in a neutron flux.

Supplementary to the slurry program to be carried out under Chemical Reprocessing Research and Development, two forced circulation Bi-Th_3-Bi_5 slurry loops will be constructed to determine the corrosive and erosive effects of the slurry on graphite and Croloy; the transportability of the thorium-bismuthide particles, and the stability of thorium-bismuthide particle size under thermal gradients.

B. Reference Parameters

To expedite the formulation of a practical Research and Development Program it was necessary to establish certain conditions under which the required tests should be run. These parameters, including maximum and minimum temperature, temperature gradients, velocity, and materials, were selected to obtain the most valuable data in a minimum of time, and, for the most part, are based on research performed at Brookhaven National Laboratory.

Selection of a container material is based on results of extensive corrosion testing in which a wide variety of materials was investigated. Maximum corrosion resistance was found in the low-chromium-molybdenum steels and the majority of subsequent corrosion testing was performed on Croloy 2 1/4 due to its excellent elevated temperature properties. Croloy 1 1/4 will be investigated as an alternate material because of its ease of fabrication, reduced cost, comparable mechanical properties and expected good corrosion behavior at proposed operating conditions.

The minimum temperature is governed by the solubility limit of uranium in bismuth and should be as low as possible in order to obtain the greatest temperature gradient compatible with materials of construction. The solubility of uranium in liquid bismuth increases with increasing temperature, on the order of 1100 ppm at 660 F, 2000 ppm at 750 F and 3500 ppm at 840 F. Fission density requirements and fission product poisoning effects dictate a minimum concentration of 1500 ppm. On this basis, a minimum bulk with a film temperature of 720 F is proposed, thus allowing an operating margin of 200 ppm or 15 F.

The mass transfer and corrosion resistance of Croloy 2 1/4 in a liquid bismuth system are dependent upon the maximum temperature of the bismuth and the temperature gradient across the system. Data from Brookhaven indicate that Croloy 2 1/4 may suffer considerable corrosion and mass transfer at temperature differentials above 180 F with a minimum temperature of 750 F. Increasing both the minimum and maximum temperatures would tend to produce accelerated corrosion. On the other hand, corrosion loops operated at gradients of 75 F and 160 F exhibit less mass transfer and indicated that Croloy 2 1/4 should be satisfactory for use as a container material in a liquid bismuth-uranium system operating from 750 F to 910 F.

Preliminary tests show that Croloy 2 1/4 is subject to mass transfer in the reference bismuth-uranium fuel solution with a gradient as low as 70 F; it is evident, therefore, that mass transfer inhibitors must be added to the system if low alloy steels are to be used. Based on corrosion studies with other heavy liquid metal, metallic inhibitors Zr and Ti were considered. Zirconium has the lower thermal neutron capture cross section and, by virtue of its being a product of uranium fission, will be constantly produced in the fuel stream. Furthermore, it is reported that the reaction of graphite with liquid bismuth-uranium is inbibited by a Zr addition, and it is hoped that similar successful inhibition of mass transfer of low chromium steel container will be accomplished.

Subsequent corrosion testing, using Zr in concentrations up to 350 ppm as an additive to the bismuth-uranium fluid, shows that at least partial inhibition of both dissolution and precipitation of Fe in Bi is achieved. Experimental data suggest that the action of Zr as an inbibitor may lie in its ability to form a protective deposit on the steel surface. Further experiment and examination give some credence to the theory that this deposit may be an adherent film of zirconium nitride, formed by the reaction of the Zr with the nitrogen inherently present in the steel.

Recent experience at BNL indicates that the nature of heat treatment given the steel prior to contact with molten bismuth-uranium might substantially affect its corrosion resistance and mass transfer properties. Work reported in a chemical paper to the German Ironworks

Association in 1948* indicated the existence of a certain relationship between heat treating temperatures and amount of nitrides found in solid solution within the steel. Heating beyond 930 F precipitated stable nitrides; and subsequent quenching of the steel from temperatures around 1800 F was required to restore the solid solution.

The theory of the formation of the ZrN protective film is predicated on the reaction of the zirconium additive with free or dissolved nitrogen within the steel. Hence, maintenance of the steel at temperatures approaching 930 F, thereby precipitating stable nitrides, could prove deleterious to its corrosion resistance.

The proposed maximum operating temperature of 885 F is selected primarily on the basis of the successful operation of corrosion loops at Brookhaven. If the ZrN theory is proven and the relationship of available nitrogen to temperature exists, a maximum temperature of 885 F would permit successful operation of the system. It is felt that the resulting temperature difference of 135 F approaches the minimum required for the economic operation of a LMFR.

To the end of exploring a larger temperature differential compatible with the proposed materials of construction, an alternate set of temperature conditions is proposed. Under these conditions a temperature gradient of 225 F will be obtained by raising the maximum temperature to 975 F. Most of the information

* Kempf, H. and Abresch, K., "The Acid Solubility and Electrolytic Decomposition of Nitrides Occurring in Alloyed and Unalloyed Steels", Arch Eisenhuttenw, 11-12, May-June, pp 261-270 (1944).

resulting from Brookhaven corrosion testing indicates that this temperature differential would be too high from a mass transport standpoint. In a few cases, particularly those involving forced circulation corrosion loops, Croloy 2 1/4 has shown satisfactory resistance for a short period of time at high gradients.

Selection of velocity parameters has limited basis. The forced circulation loop at BNL was operated with velocities varying from 4 fps to 8 fps and indicate no difference in corrosion and mass transfer rates. Velocities of 8 fps to 10 fps are proposed since some data is available concerning bismuth corrosion at these values. The effect of decreased velocity will be checked in testing at 4 fps to 5 fps. To determine the effect of extra low velocity as required in low power reactor operation, a corrosion test will be performed in the utility loop with a flow velocity of 1 fps.

C. Forced Cirdulation Loop Corrosion Test Program

The following program is designed to provide the minimum of information necessary to confirm Croloy 2 1/4 and/or Croloy 1 1/4 as suitable materials of construction for the LMFRE. Conditions and parameters as proposed herein are subject to modification or additions which may be dictated by the outcome of supporting investigations preceding or accompanying the forced circulation tests.

Prior to operation at proposed testing conditions, all loops will be preconditioned by the addition of 350 ppm Zr and 250 ppm Mg unless otherwise specified. In all loops uranium concentration will be 1500 ppm. During loop operation, close control of flow and temperature conditions will be maintained and pump performance will be recorded.

Sampling the fluid to determine concentrations of additives and uranium will be performed regularly and periodic radiography of the test sections will be made to observe any corrosion and/or precipitation. Corrosion will be determined both qualitatively and quantitatively.

1. DYNAMIC UTILITY TEST LOOP (TEST #1)

Objectives:

a. To determine the suitability of various pumps for use in other loops.

b. To test graphite to graphite and graphite to metal joints, valves, expansion joints, etc.

c. For use in substantiating test results of alternate supporting tests.

d. To determine effect of extra-low velocity (1 fps) on corrosion and mass transfer.

Conditions and Parameters:

a. Material of construction: Croloy 2 1/4

b. Loop size: 3/4" schedule 40 ips

c. Test fluid: U-Bi with addition of Mg & Zr

d. Temperatures: maximum 887 F, Δ T 135F

e. Flow: variable

f. Heat treatment: annealed

g. Test duration: various

2. TESTING OF CROLOY 2 1/4 (REFERENCE CONDITIONS)
(A) DEMONSTRATION LOOPS (TEST #2 AND #3)

Objectives:

a. To demonstrate the feasibility of the selected design parameters.

b. To determine the corrosion resistance of Croloy 2 1/4 base metal and weld metal in fuel solutions under the specified operating conditions.

Conditions and Parameters:

a. Material of construction: Croloy 2 1/4

b. Loop size: 3/4" schedule 40 ips

c. Test fluid: U-Bi with addition of Mg and Zr

d. Temperatures: maximum 887 F, Δ T 135 F

e. Flow: 8 to 10 fps

f. Test duration: 10, 000 hrs.

g. Heat treatment: annealed

(B) CORROSION RATE DETERMINATION (TEST #4A, #4B, and #4C)

Objective:

a. To determine a corrosion rate for Croloy 2 1/4 under the specified test conditions.

Conditions and Parameters:

a. Material of construction: Croloy 2 1/4

b. Loop size: 3/4" schedule 40 ips

c. Test fluid: U-Bi with addition of Mg and Zr

d. Temperatures: maximum 887 F, Δ T 135 F

e. Flow: 8 to 10 fps

f. Heat treatment: annealed

g. Test duration:*

4A - 1500 hrs.

4B - 3000 hrs.

4C - 5500 hrs.

* Corrosion rates will be determined from samples taken from the loop at 1500, 3000 and 5500 hours.

(C) VELOCITY EFFECT (TEST #5E and #5F)

Objective:

a. To determine the effect of velocity on the corrosion of Croloy 2 1/4 in fuel solutions under the specified test conditions.

Conditions and Parameters:

a. Material of construction: Croloy 2 1/4

b. Loop size: 3/4" schedule 40 ips

c. Test fluid: U-Bi with additions of Mg and Zr.

d. Flow: 4 to 5 fps

e. Temperatures: maximum 887 F, Δ T 135 F

f. Heat treatment: annealed

g. Test duration:

5E - 1500 hrs.

5F - 1500 hrs.

(D) ADDITIVES (TEST #5C and #5D)

Objective:

a. To determine the effect of additives on the corrosion of Croloy 2 1/4 in fuel solutions under the specified test conditions.

Conditions and Parameters:

a. Material of construction: Croloy 2 1/4

b. Loop size: 3/4" schedule 40 ips

c. Test fluid: U-Bi with additions to be determined from alternate supporting tests.

d. Temperatures: maximum 887 F, Δ T 135 F

e. Flow: 8 to 10 fps

f. Test duration:

5C - 1500 hrs.

5D - 1500 hrs.

g. Heat treatment: annealed

(E) HEAT TREATMENT EFFECT (TEST #5A and #5B)

Objective:

a. To determine the effect of heat treatment on the corrosion of Croloy 2 1/4 under the specified test conditions.

Conditions and Parameters:

a. Material of construction: Croloy 2 1/4

b. Loop size: 3/4" schedule 40 ips

c. Test fluid: U-Bi with addition of Mg and Zr

d. Flow: 8 to 10 fps

e. Temperatures: maximum 887 F, ΔT 135 F

f. Heat treatment: normalized and tempered

g. Test duration:

5A - 1500 hrs.

5B - 1500 hrs.

3. TESTING OF CROLOY 2 1/4 (ALTERNATE CONDITIONS)
(A) DEMONSTRATION LOOPS (TEST #6 AND #7)

Objectives:

a. To demonstrate the feasibility of the selected design parameters.

b. To determine the corrosion resistance of Croloy 2 1/4 base metal and weld metal in fuel solutions under the specified operating conditions.

Conditions and Parameters:

a. Material of construction: Croloy 2 1/4

b. Loop size: 3/4" schedule 40 ips

c. Test fluid: U-Bi with addition of Mg and Zr

d. Temperatures: maximum 977 F, Δ T 135 F

e. Flow: 8 to 10 fps

f. Test duration: 10,000 hrs.

g. Heat treatment: annealed

(B) CORROSION RATE DETERMINATION (TEST #8A, #8B and #8C)

Objective:

a. To determine a corrosion rate for Croloy 2 1/4 under the specified test conditions.

Conditions and Parameters:

a. Material of construction: Croloy 2 1/4

b. Loop size: 3/4" schedule 40 ips

c. Test fluid: U-Bi with addition of Mg and Zr

d. Temperatures: maximum 977 F, Δ T 225 F

e. Flow: 8 to 10 fps

f. Heat treatment: annealed

g. Test duration:*

8A - 1500 hrs.

8B - 3000 hrs.

8C - 5500 hrs.

* Corrosion rates will be determined from samples taken from the loop at 1500, 3000 and 5500 hours.

(C) VELOCITY EFFECT (TEST #9E and #9F)

Objective:

a. To determine the effect of velocity on the corrosion of Croloy 2 1/4 in fuel solutions under the specified test conditions.

Conditions and Parameters:

a. Material of construction: Croloy 2 1/4

b. Loop size: 3/4" schedule 40 ips

c. Test fluid: U-Bi with addition of Mg and Zr

d. Flow: 4 to 5 fps

e. Temperatures: maximum 977 F, ΔT 225 F

f. Heat treatment: annealed

g. Test duration:

9E - 1500 hrs.

9F - 1500 hrs.

(D) ADDITIVES (TEST #9C and #9D)

Objective:

a. To determine the effect of additives on the corrosion of Croloy 2 1/4 in fuel solutions under the specified test conditions.

Conditions and Parameters:

a. Material construction: Croloy 2 1/4

b. Loop size: 3/4" schedule 40 ips

c. Test fluid: U-Bi with additions to be determined from alternate supporting tests.

d. Temperatures: maximum 977 F, Δ T 225 F

e. Flow: 8 to 10 fps

f. Test duration:

9C - 1500 hrs.

9D - 1500 hrs.

g. Heat treatment: annealed

(E) HEAT TREATMENT EFFECT (TEST #9A and #9B)

Objective:

a. To determine the effect of heat treatment on the corrosion of Croloy 2 1/4 under the specified test conditions.

Conditions and Parameters:

a. Material of construction: Croloy 2 1/4

b. Loop size: 3/4" schedule 40 ips

c. Test fluid: U-Bi with addition of Mg and Zr

d. Flow: 8 to 10 fps

e. Temperatures: maximum 977 F, ΔT 225 F

f. Heat treatment: normalized and tempered

g. Test duration:

9A - 1500 hrs.

9B - 1500 hrs.

4. TESTING OF CROLOY 1 1/4 (REFERENCE CONDITIONS)

(A) DEMONSTRATION LOOPS (TEST #10 AND #11)

Objectives:

a. To demonstrate the feasibility of the selected design parameters.

b. To determine the corrosion resistance of Croloy 1 1/4 base metal and weld metal in fuel solutions under the specified operating conditions.

Conditions and Parameters:

a. Material of construction: Croloy 1 1/4

b. Loop size: 3/4" schedule 40 ips

c. Test fluid: U-Bi with addition of Mg and Zr

d. Temperatures: maximum 887 F, Δ T 135 F

e. Flow: 8 to 10 fps

f. Test duration: 10, 000 hrs.

g. Heat treatment: annealed

(B) CORROSION RATE DETERMINATION (TEST #12A, #12B and #12C)

Objective:

a. To determine a corrosion rate for Croloy 1 1/4 under the specified test conditions.

Conditions and Parameters:

a. Material of construction: Croloy 1 1/4

b. Loop size: 3/4" schedule 40 ips

c. Test fluid: U-Bi with addition of Mg and Zr

d. Flow: 8 to 10 fps

e. Temperatures: maximum 887 F. Δ T 135 F

f. Heat treatment: annealed

g. Test duration:*

12A - 1500 hrs.

12B - 3000 hrs.

12C - 5500 hrs.

* Corrosion rates will be determined from samples taken from the loop at 1500, 3000 and 5500 hrs.

(C) VELOCITY EFFECT (TEST #13E and #13F)

Objective:

a. To determine the effect of velocity on the corrosion of Croloy 1 1/4 in fuel solutions under the specified test conditions.

Conditions and Parameters:

a. Material of construction: Croloy 1 1/4

b. Loop size: 3/4" schedule 40 ips

c. Test fluid: U-Bi with addition of Mg and Zr

d. Flow: 4 to 5 fps

e. Temperatures: maximum 887 F, ΔT 135 F

f. Heat treatment: annealed

g. Test duration:

13E - 1500 hrs.

13F - 1500 hrs.

(D) ADDITIVES (TEST #13C and #13D)

Objective:

a. To determine the effect of additives on the corrosion of Croloy 1 1/4 in fuel solutions under the specified test conditions.

Conditions and Parameters:

a. Material of construction: Croloy 1 1/4

b. Loop size: 3/4" schedule 40 ips

c. Test fluid: U-Bi with additions to be determined from alternate supporting tests.

d. Temperatures: maximum 887 F, ΔT 135 F

e. Flow: 8 to 10 fps

f. Test duration:

13C - 1500 hrs.

13D - 1500 hrs.

g. Heat treatment: annealed

(E) HEAT TREATMENT EFFECT (TEST #13A and #13B)

Objective:

a. To determine the effect of heat treatment on the corrosion of Croloy 1 1/4 under the specified test conditions.

Conditions and Parameters:

a. Material of construction: Croloy 1 1/4

b. Loop size: 3/4" schedule 40 ips

c. Test fluid: U-Bi with addition of Mg and Zr

d. Flow: 8 to 10 fps

e. Temperatures: maximum 887 F, Δ T 135 F

f. Heat treatment: normalized and tempered

g. Test duration:

13A - 1500 hrs.

13B - 1500 hrs.

5. TESTING OF CROLOY 1 1/4 (ALTERNATE CONDITIONS)
(A) DEMONSTRATION LOOPS (TEST #14 AND #15)

Objectives:

a. To demonstrate the feasibility of the selected design parameters.

b. To determine the corrosion resistance of Croloy 1 1/4 base metal and weld metal in fuel solutions under the specified operating conditions.

Conditions and Parameters:

a. Material of construction: Croloy 1 1/4

b. Loop size: 3/4" schedule 40 ips

c. Test fluid: U-Bi with addition of Mg and Zr

d. Temperatures: maximum 977 F, Δ T 225 F

e. Flow: 8 to 10 fps

f. Test duration: 10, 000 hrs.

g. Heat treatment: annealed

(B) CORROSION RATE DETERMINATION (TEST #16A, #16B and #16C)

Objective:

a. To determine a corrosion rate for Croloy 1 1/4 under the specified test conditions.

Conditions and Parameters:

a. Material of construction: Croloy 1 1/4

b. Loop size: 3/4" schedule 40 ips

c. Test fluid: U-Bi with addition of Mg and Zr

d. Flow: 8 to 10 fps

e. Temperatures: maximum 977 F, ΔT 225 F

f. Heat treatment: annealed

g. Test duration: *

16A - 1500 hrs.

16B - 3000 hrs.

16C - 5500 hrs.

* Corrosion rates will be determined from samples taken from the loop at 1500, 3000 and 5500 hrs.

(C) VELOCITY EFFECT (TEST #17E and #17F)

Objective:

a. To determine the effect of velocity on the corrosion of Croloy 1 1/4 in fuel solutions under the specified test conditions.

Conditions and Parameters:

a. Material of construction: Croloy 1 1/4

b. Loop size: 3/4" schedule 40 ips

c. Test fluid: U-Bi with addition of Mg and Zr

d. Flow: 4 to 5 fps

e. Temperatures: maximum 977 F, ΔT 225 F

f. Heat treatment: annealed

g. Test duration:

17E - 1500 hrs.

17F - 1500 hrs.

(D) ADDITIVES (TEST #17C and #17D)

Objective:

a. To determine the effect of additives on the corrosion of Croloy 1 1/4 in fuel solutions under the specified test conditions.

Conditions and Parameters:

a. Material of construction: Croloy 1 1/4

b. Loop size: 3/4" schedule 40 ips

c. Test fluid: U-Bi with additions to be determined from alternate supporting tests.

d. Temperatures: maximum 977 F, ΔT 225 F

e. Flow: 8 to 10 fps

f. Test duration:

17C - 1500 hrs.

17D - 1500 hrs.

g. Heat treatment: annealed

(E) HEAT TREATMENT EFFECTS (TEST #17A and #17B)

Objective:

a. To determine the effect of heat treatment on the corrosion of Croloy 1 1/4 under the specified test conditions.

Conditions and Parameters:

a. Material of construction: Croloy 1 1/4

b. Loop size: 3/4" schedule 40 ips

c. Test fluid: U-Bi with addition of Mg and Zr

d. Flow: 8 to 10 fps

e. Temperatures: maximum 977 F, ΔT 225 F

f. Heat treatment: normalized and tempered

g. Test duration:

17A - 1500 hrs.

17B - 1500 hrs.

6. SLURRY LOOPS (TEST #18 and #19)

Objectives:

a. To determine the corrosive and erosive effects of a $Bi-Th_3Bi_5$ slurry on graphite and metals.

b. To determine the transportability of thorium-bismuthide slurries.

c. To determine the stability of particle size with temperature differentials.

Conditions and Parameters:

a. Material of construction: Croloy 2 1/4

b. Loop size: 3/4" schedule 40 ips

c. Test fluid: Th_3Bi_5 in Bi

d. Flow: to be determined

e. Temperatures: maximum 885 F, ΔT 135 F

f. Test duration: 5,000 hrs.

7. CAUSTIC STRESS CORROSION OF CROLOY 2 1/4 (Test #20)

Objective:

To determine the susceptibility of Croloy 2 1/4 in boiling water to stress induced caustic embrittlement.

Conditions and Parameters:

1. Test material: 2 1/4 Cr-1 Mo steel (Croloy 2 1/4)
2. Test fluid: boiling water with the following quality specifications:
   - a. O_2 0.005 ppm (max.)
   - b. SiO_2 0.02 ppm (max.)
   - c. Cl 0.01 ppm (max.)
   - d. Total dissolved solids 0.25 ppm (max.)
   - e. pH adjusted with ammonia to maintain the following concentrations

     Fe 0.01 ppm (max.)

     Cu 0.005 ppm (max.)
   - f. Hardness

     Cu zero
   - g. NaOH added in the following amounts
     1) 0.001%
     2) 0.01%
     3) 0.1%
     4) 1.0%
     5) 10%
     6) 35%

3. Pressure: 600 psi

4. Temperature (saturation): 486 F

8. DYNAMIC CORROSION TESTING OF CROLOY 2 1/4 IN BOILING WATER (Test #21)

Objective:

To determine the corrosion resistance of Croloy 2 1/4 in boiling water so that its suitability for use as a material of construction for the LMFRE once-through steam generator may be accurately evaluated.

Conditions and Parameters:

1. Test material: 2 1/4 Cr-1 Mo steel (Croloy 2 1/4)

2. Test fluid: boiling water with the following quality specifications:

   a. O_2 0.005 ppm (max.)

   b. SiO_2 0.02 ppm (max.)

   c. Cl 0.01 ppm (max.)

   d. Total dissolved solids 0.25 ppm (max.)

   e. pH adjusted with ammonia to maintain the following concentrations

      Fe 0.01 ppm (max.)

      Cu 0.005 ppm (max.)

   f. Hardness

      Cu zero

3. Pressures and corresponding saturation temperatures

   a. 550 psi - 477 F

   b. 600 psi - 486 F

   c. 650 psi - 495 F

   d. 700 psi - 503 F

4. Flow velocity

   a. 1 fps

   b. 2 fps

   c. 3 fps

D. Supporting Tests and Investigations

This program is designed to provide basic information which can be applied to the forced-circulation corrosion test loops, and to obtain supplementary material data applicable to the LMFR concept. Testing is based on twenty tilting autoclave facilities with which desired conditions of heat treatment, temperative gradient and fluid composition can be achieved.

Three autoclaves will be run to determine a duration from which significant corrosion data can be derived.

Analysis of the fuel solution will be as follows unless otherwise specified:

Uranium - 1500 ppm
Zirconium - 350 ppm
Magnesium - 250 ppm
Bismuth - Balance

Because of certain restrictions such as alternate wetting and drying of the test samples, impracticality of pre-conditioning, and lack of continuous flow past the samples, information supplied by the tilting autoclaves will be considered qualitative and further testing will subsequently be proposed where the need for quantitative data is established.

In addition to the tilting autoclave tests, various static pot-type tests will be performed to study materials compatibility in liquid bismuth.

In certain areas where technology is not sufficiently advanced to propose specific tests, preliminary investigations will be performed. Such studies include development of control rod materials, selection of materials for chemical processing equipment, development of slurry technology, and investigation of the ZrN protective film theory.

1. HEAT TREATMENT EFFECT (Test A - Tilting Autoclave)

Objectives:

a. To study factors affecting the formation and stability of the protective film and the resulting effect on the the corrosion and erosion resistance of Croloy 2 1/4 and Croloy 1 1/4.

b. To determine the effect of heat treatment on the corrosion resistance of Croloy 2 1/4 and Croloy 1 1/4.

Conditions and Parameters:

a. Test materials: Croloy 2 1/4
   Croloy 1 1/4

b. Heat treatment: annealed at 1575 F
   normalized at 1700 F and tempered at 1300 F
   normalized at 1700 F and tempered at 975 F

c. Test fluid: U-Bi with additives

d. Temperatures: maximum 975 F, Δ T 225 F

2. ADDITIVE PERFORMANCE (Test B - Tilting Autoclave)

Objectives:

a. To study the effectiveness of Mg, Th and Ca as oxygen getters.

b. To study factors affecting the formation and stability of the zirconium nitride protective film and the resulting effect on the corrosion and erosion resistance of Croloy 2 1/4.

c. To determine the effectiveness of Zr and Ti as inhibitors.

d. To determine the effect of U concentration on corrosion resistance of Croloy 2 1/4.

Conditions and Parameters:

a. Test material: Croloy 2 1/4

b. Test fluid: U-Bi, varying amounts and types of additives

c. Additives: uranium - 1,000, 1,500, 2,000 ppm
   oxygen getters - Mg 250, 500, 1,000 ppm
   Ca 250, 500, 1,000 ppm
   Th 250, 500, 1,000 ppm
   inhibitors - Zr 250, 500, 1,000 ppm
   Ti 250, 500, 1,000 ppm

d. Temperatures: maximum 975 F, Δ T 225 F

3. CLEANLINESS EFFECT (Test C - Tilting Autoclave)

Objective:

a. To determine the effect of cleanliness on the corrosion and mass transfer of Croloy 2 1/4 in liquid Bi.

Conditions and Parameters:

a. Test materials: Croloy 2 1/4

b. Test fluid: U-Bi additives

c. Temperature: maximum 975 F, Δ T 225 F

d. Methods of cleaning: commercial (fused salt)
sand blast
chemical (acid)

4. FISSION PRODUCTS EFFECT (Test D - Tilting Autoclave)

Objective:

To determine the effect of fission products on the corrosion resistance of Croloy 2 1/4 in bismuth.

Conditions and Parameters:

a. Test material: Croloy 2 1/4

b. Test fluid: U-Bi $\pm$ additives $\pm$ "misch metal" (rare earths)
U-Bi $\pm$ additives $\pm$ noble metals (Ag, Sb, Ru)

c. Temperatures: maximum 975 F, Δ T 225 F

5. CORROSION PRODUCTS EFFECT (Test E - Tilting Autoclave)

Objective:

To determine the effect of corrosion products on the corrosion resistance of Croloy 2 1/4 in liquid bismuth.

Conditions and Parameters:

a. Test material: Croloy 2 1/4

b. Test fluid: U-Bi $\pm$ additives saturated with Croloy corrosion products.

c. Temperatures: maximum 975 F, Δ T 225 F

6. WETTING CHARACTERISTICS (Test F - Tilting Autoclave)

Objective:

To observe the effect of various surface conditions on the wetting characteristics of Croloy 2 1/4 in liquid bismuth.

Conditions and Parameters:

a. Test material: Croloy 2 1/4

b. Test fluid: U-Bi $\pm$ additives

c. Temperatures: maximum 975 F, Δ T 225 F

d. Surface condition:

1) strongly oxidized

2) hydrogen saturated by pickling

3) distorted (honed)

4) undistorted (lightly ground)

5) irregular (sand blasted)

6) artificially wet (bismuth plated or shot blasted with steel shot and bismuth pellets)

7. WELDING CONDITIONS (Test G - Tilting Autoclave)

Objective:

To study the effects of welding conditions on the corrosion resistance of Croloy 2 1/4.

Conditions and Parameters:

a. Test material: Croloy 2 1/4 welded joint

b. Test fluid: U-Bi ± additives

c. Temperatures: maximum 975 F, Δ T 225 F

d. Welding conditions

A. Coated electrode welded

1) as welded
2) stress relieved at 1300 F
3) stress relieved at 975 F
4) normalized at 1700 F
5) normalized, drawn at 1350 F
6) normalized, drawn at 975 F

B. Inert gas welded

1) as welded
2) stress relieved at 1300 F
3) stress relieved at 975 F
4) normalized at 1700 F
5) normalized, drawn at 1300 F
6) normalized, drawn at 975 F

8. STRESS RUPTURE TESTING IN FUEL SOLUTIONS (Test H)

<u>Objective:</u>

To determine the effect of fuel solutions upon the stress-rupture properties of reference materials at operating temperatures.

<u>Conditions and Parameters:</u>

a. Test materials: Croloy 2 1/4
   Croloy 1 1/4

b. Test fluid: U-Bi ± additives

c. Temperatures: 885 F
   975 F

Ten-thousand-hour-stress-rupture curves will be determined for each material at 885 F and 975 F.

9. ELECTROCHEMICAL EFFECT (Test I)

Objective:

To determine the effect of an externally applied electrical potential on the corrosion resistance of Croloy 2 1/4 in liquid Bi.

Conditions and Parameters:

a. Test materials: Croloy 2 1/4

b. Test fluid: high purity Bi (99.999)

C.P. Bi

c. Temperature: 975 F

Croloy electrodes will be held at potentials negative to, equal to, and positive to liquid Bismuth, and the relative corrosion rates of the three electrodes will be observed.

10. STATIC CORROSION TEST (Test J)

Objectives:

a. To confirm the corrosion resistance of tantalum, beryllium, and molybdenum to liquid bismuth.

b. To investigate the stability of these materials in liquid bismuth at proposed reactor operating temperatures.

Conditions and Parameters:

a. Test materials: tantalum
molybdenum alloy (99.5% Mo-0.5% Ti)
beryllium

b. Test fluid: U-Bi ± additives

c. Temperature: 975 F

11. INVESTIGATION OF NITROGEN AVAILABILITY IN LOW CHROME STEELS AND THE RESULTING EFFECT ON THE FORMATION OF A ZrN FILM (Study K)

a. Random heats of Croloy 2 1/4 and Croloy 1 1/4 from the B&W Tube Division, Beaver Falls, Pa. will be analyzed to determine the range of available nitrogen contained in this material. Samples from these heats will be water quenched from 1800 F and analyzed for:

Total Al (including Al_2O_3)

Acid soluble Al

Acid soluble N

Acid insoluble N

The heats from which other materials testing equipment will be constructed will be analyzed for these constituents in the as-received condition.

b. To determine the effect of heat treatment, available nitrogen will be determined for samples in the following conditions:

Normalized at 1700 F

Normalized at 1700 F and tempered at 975 F for 100 hours

Normalized at 1700 F and tempered at 885 for 100 hours

Fully annealed

Fully annealed and reheated to 885 F for 100 hours

Fully annealed and reheated to 975 F for 100 hours

c. The effect of time at temperature will also be determined by analyzing for available nitrogen in samples held at tempering temperature for varying lengths of time.

12. INVESTIGATION OF CORROSION AND MASS TRANSFER INHIBITION BY ZrN FILM FORMATION (Study L)

Objective:

The following experiments will be performed to determine the feasibility of corrosion and mass transfer inhibition through formation of a protective layer of zirconium nitride.

a. Special vacuum-melted high purity alloys of Croloy 2 1/4 in liquid Bi-U-Zr solution will be subjected to static exposure tests and dynamic tilting-capsule corrosion tests to determine the existence of a ZrN film. The tests will be conducted with and without both nitrogen and carbon to exclude the possibility of Zr-C or ZrN film formation. If a protective film forms it will be identified by metallographic means and X-ray or electron diffraction analysis.

b. The minimum nitrogen content required to form and maintain the ZrN film will be determined as soon as the feasibility of its formation is verified.

c. The effects of other alloying elements in Croloy 2 1/4 (Mn, Mo, Si) which combine with nitrogen during ZrN film formation will be investigated. Vacuum melted alloys of Fe, C, and Cr with varying concentrations of the additives, Mn, Mo, and Si,will be tested to accomplish this objective.

d. Studies will be carried out to determine the solubility and corrosion resistance of pure ZrN in liquid bismuth solutions. ZrN will be in the form of solid-test specimens.

e. Possible corrosion-inhibition by Ti-N film formation will be investigated.

13. INVESTIGATION OF THE MECHANICAL PROPERTIES OF CROLOY 2 1/4 (Study M)

Consideration of nitrogen availability for protective film formation and repairs by remote control may require unconventional heat treatments; e.g., normalizing, followed by tempering at a temperature below the usual post weld treatments. Therefore, the effects of such treatments upon the mechanical properties of Croloy 2 1/4 will be invéstigated.

The following properties will be determined:

a) Tensile, hardness, impact and micro-structure of thin and heavy sections of Croloy 2 1/4

1) normalized from above the A_3 point

2) normalized and tempered at 900 F, 1000 F, 1100 F, 1200 F and 1300 F respectively for 1/2, 1, 2 and 4 hours.

b) Transverse weld tensile, bend, hardness, and micro-structure of weldments made with normalized and tempered Croloy 2 1/4.

1) argon-arc or helium-arc welded with a base metal filler material, welded and post weld treated at 900 F, 1000 F, 1200 F, 1300 F, respectively.

2) arc welded with standard coated electrodes and post weld treated at 900 F, 1000 F, 1100 F, 1200 F and 1300 F.

14. STATIC TEST POTS (Test N)

Objective:

To determine the compatibility of various thermal insulations with liquid Bi.

Conditions and Parameters:

a. Material of construction: Croloy 2 1/4

b. Temperature: 975 F

c. Test fluid: Bi

d. Test samples: various promising insulation materials.

15. SELECTION OF COMPATIBLE MATERIALS FOR SERVICE IN THE CHEMICAL PROCESSING PLANT (Study O)

Objective:

To determine what materials of construction can be used for the chemical processing system.

16. SLURRY TECHNOLOGY DEVELOPMENT (Study P)

An important facet of the Liquid Metal Fuel Reactor concept is the breeder blanket, consisting of a dispersion of particles of thorium-bismuth inter-metallic in molten bismuth. The development of Th-Bi slurries was initiated at Brookhaven and additional work on the constitution of the dispersion was performed by Horizons, Inc., of Cleveland, Ohio.

While the LMFRE will not include a blanket as such, blanket tests will be accomplished by subjecting forced circulation slurry loops to neutron irradiation in test holes in the LMFRE reflector.

Before such tests can be instigated with a reasonable guarantee of obtaining valid in-pile data, the compatibility of container materials and the practicability of pumping the slurry must be demonstrated.

In achievement of these objectives, dynamic corrosion tests imposing the following conditions should be employed:

a. Test duration: 3000 hrs.

b. Material of construction: Croloy 2 1/4

c. Pipe size: 3/4 inch (40) ips

Minimum Program

a. Two tests with velocity 3 fps

Max. temp. 885 F

Δ T 135 F

b. One test with velocity 3 fps

Max. temp. 975 F

Δ T 225 F

c. One test with velocity 5 fps

Max. temp. 885 F

Δ T 135 F

In addition, the minimum velocity necessary to maintain the dispersion must be determined since in reactor operation, flow of the blanket may approach values as low as 0.003 ft/sec.

17. STATIC POT TESTS (Test Q)

Objective:

To determine the out-gassing characteristics of graphite.

Conditions and Parameters:

a. Material of construction: Croloy 2 1/4

b. Temperature: variable

c. Test fluid: U-Bi

d. Test pressure

Pots will be leak tight and evacuated so that the out-gassing characteristics of graphite can be studied with variations in temperature.

18. GRAPHITE-TO-GRAPHITE JOINING (Test R)

Objective:

To test the compatibility of a carbon-base cemented joint with liquid bismuth and investigation of possible alternate methods of joining.

Conditions and Parameters:

a. Test materials: samples of cemented graphite joints as supplied by vendors

b. Test fluid: U-Bi $\pm$ additives

c. Temperatures: 885 F
975 F

d. Pressure differential: 15 psi
25 psi
50 psi
100 psi
150 psi

Joints made by the following alternate methods may be similarly tested:

a. Pressure diffusion joint

b. Fusion weld

c. Pressure diffusion joint with metal-carbide interface layer

d. Pressure with hydrocarbon-decomposition catalyst reaction (acid-furfural)

19. INVESTIGATION OF MECHANICAL AND PHYSICAL PROPERTIES OF GRAPHITE (Study S)

Due to the wide range of properties found in varying grades and types of graphite, it is imperative that certain physical and mechanical properties of the types applicable to the LMFRE be determined.

Samples received from vendors will be tested to ascertain:

a. Density

b. Ultimate tensile strength

c. Yield strength

d. Compressive strength

e. Soundness, homogeneity

f. Thermal conductivity

g. Modulus of elasticity

h. Coefficient of expansion

20. STATIC TEST POTS----PRESSURIZED (Test T)

Objective:

To determine the depth, volume and rate of bismuth penetration into graphite.

Conditions and Parameters:

a. Test material: impervious grades of graphite supplied by vendors

b. Test fluid: Bismuth

c. Temperature: 885 F

d. Pressure differential: 15 psi
25 psi
50 psi
100 psi
150 psi

21. MOLYBDENUM PLATING OF CROLOY 2 1/4 (Study U)

In view of its excellent corrosion resistance to liquid bismuth, molybdenum may be used in the LMFRE as a protective coating for the container structure. Molybdenum chlorides are gaseous at low temperature and can be reduced to metal by hydrogen. The process involves four principal steps.

a. Contact of deoxidized Cl_2 with Mo powder at the proper temperature and partial pressure.

b. Transport of $MoCl_4$ gases to the heated region along with H_2.

c. Reduction of the $MoCl_4$ to $MoCl_2$ by H_2 on the surface to be plated.

d. Removal of the gaseous products of the reaction.

The temperature range over which reduction occurs is from 1200 F to 1500 F. The resulting plated film will be tested for integrity, resistance to temperature, and thermal cycling and corrosion resistance to liquid bismuth.

22. OUTGASSING OF GRAPHITE (Test V)

The mechanism of outgassing large samples of impervious grades of graphite will be investigated. In order that the procedures for degassing the core of the LMFRE can be accurately established, outgassing temperatures and pressures, products of the outgassing process and times necessary to reduce the volatile contaminent content to various levels will be determined.

Testing will be performed on impervious graphite samples from 6" to 24" in diameter, as received from vendors.

23. 40 INCH GRAPHITE TEST (Test W)

Due to the variation in physical and mechanical properties of graphite and the difficulties encountered in fabricating large pieces, it is not felt practical to extrapolate results obtained from screening tests of small samples. For this reason, a 40 inch cylinder of the most suitable grade of impervious graphite (as determined from the screening test program) will be subjected to a series of tests to determine physical and mechanical properties; soundness and homogeneity, and any fabrication limitations involved in producing pieces of this size.

The cylinder will be channeled prior to impregnation and procedures necessary to keep channels free of the impregnant during this process will be practiced. Characteristics of the flow channel surfaces will be determined at the completion of the impregnation operation.

To test the practicability of a built-up construction the graphite will be sectioned and re-assembled, using carbon base cemented joints. This assembly, supported with molybdenum rods, may be transported to a test facility for neutron diffusion length measurements prior to materials testing in liquid bismuth.

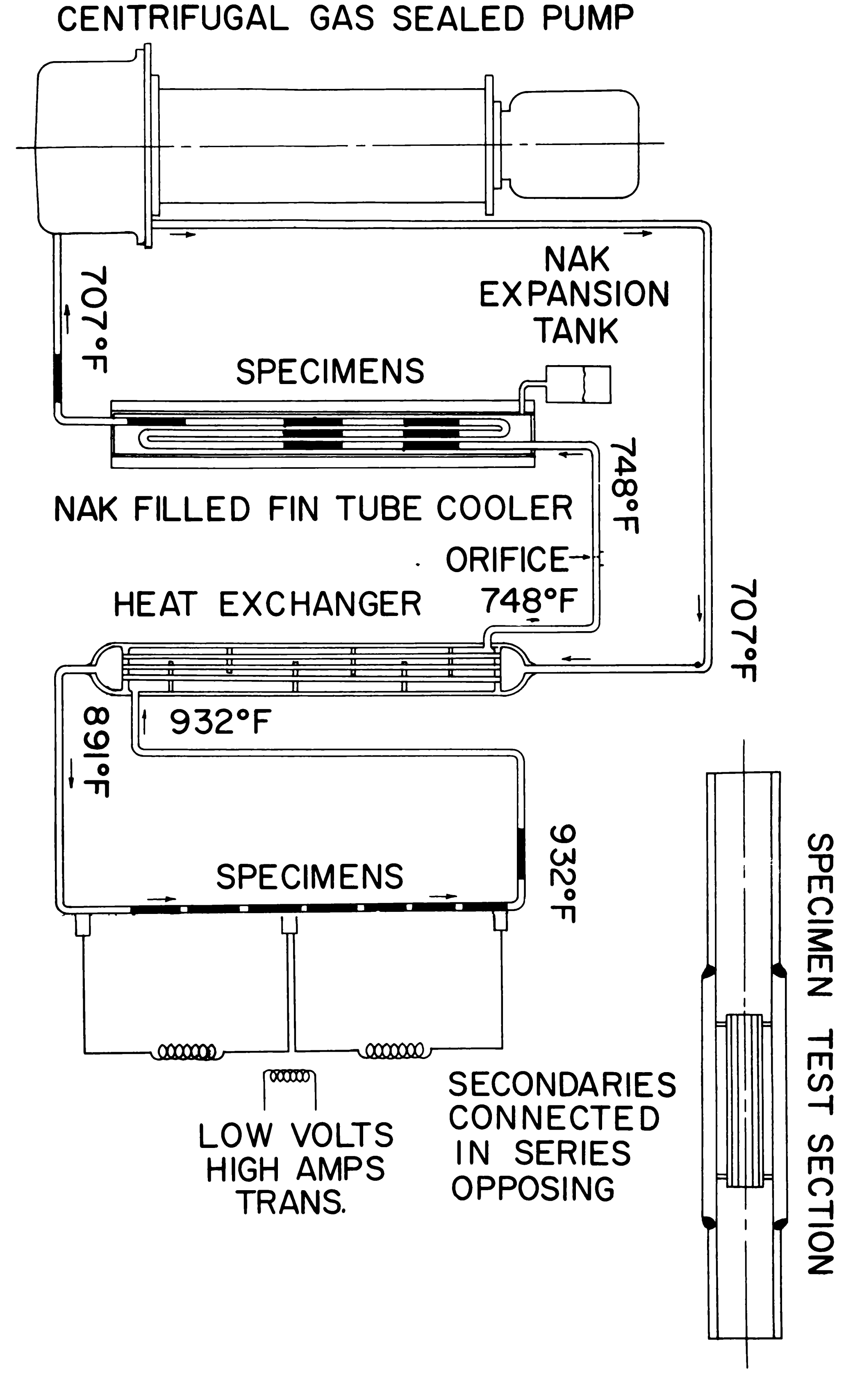
Representative samples will be cut from the graphite for determination of density, thermal conductivity, neutron absorption cross section, soundness and homogeneity.

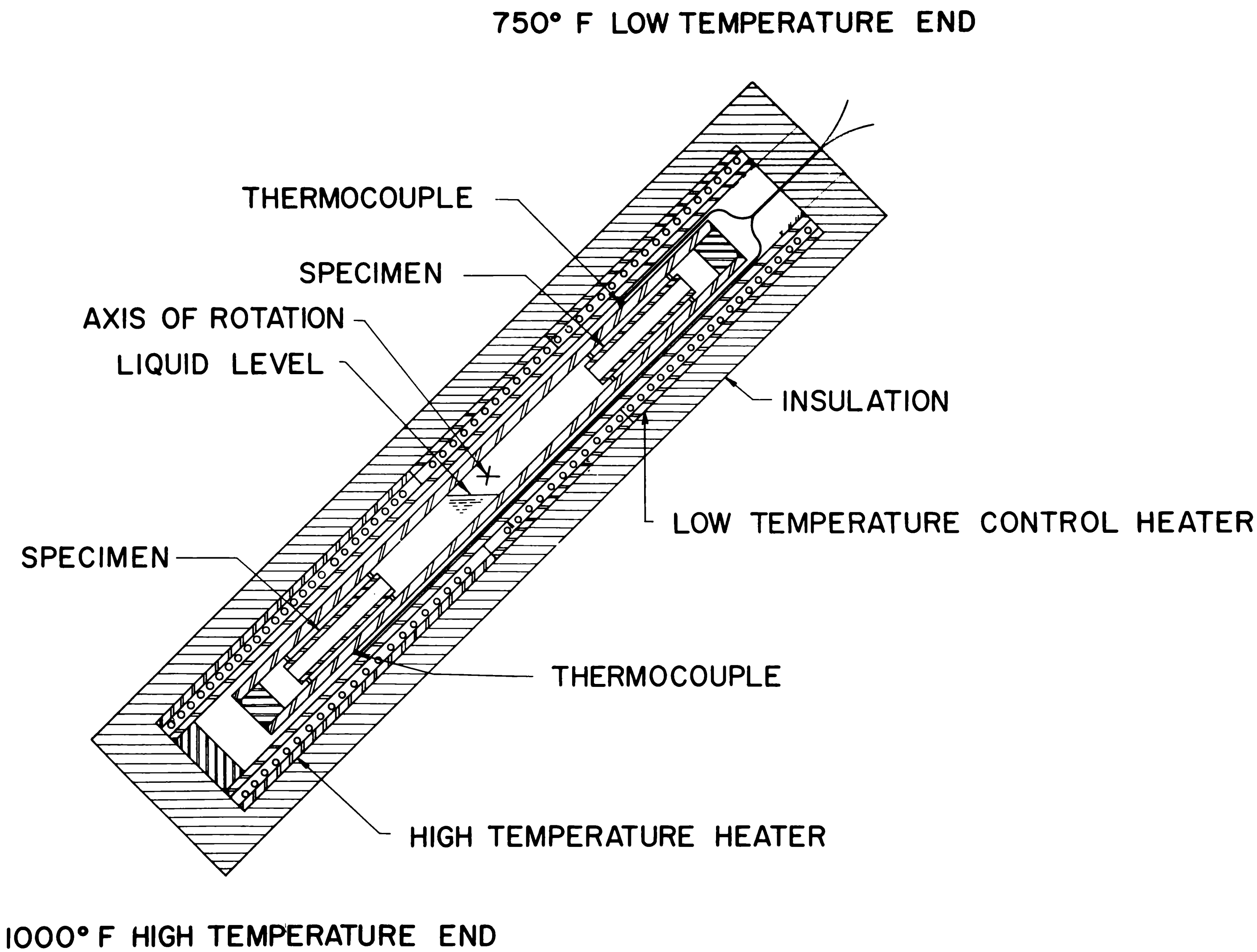
Subsequently, the 40 inch cylinder assembly will be immersed in liquid bismuth for testing to investigate bismuth-uranium penetration, uranium hold-up and other factors affecting U-Bi graphite compatability.

Practicability of a cemented joint construction and integrity of these joints as used for a large structure in a liquid bismuth system will be established.

FIGURE "8" TEST LOOP

FIGURE 2





TILTING CAPSULE APPARATUS

FIGURE 3

E. In-Pile Liquid Bismuth Corrosion Testing

In order to determine feasible materials of construction for uranium-bismuth fuel in a Liquid Metal Fuel Reactor system, it is imperative that the effects of radiation on corrosion and mechanical properties of structural materials be determined. In addition, fast neutron and fission recoil damage and fission product absorption and/or adsorption on structural materials, and the effect of fission products on the solubilities of corrosion products, uranium, and additives in uranium-bismuth must be studied.

To obtain data relating to these effects of radiation, in-pile loop studies are proposed. It is desirable that the loop studies be conducted under flux conditions approaching 10^{14}. Operation of the loops for 5000 hours is deemed necessary to obtain extrapolatable data. Regular sampling of the fluid during operation will give data concerning corrosion and fission product build up and solubilities of corrosion products, additives, and uranium in the presence of fission products in uranium-bismuth solutions. In order to obtain data from this sampling it is desirable that hot lab facilities be available. If such facilities are not available, arrangements will be made to have these analyses performed elsewhere.

Loops will contain Croloy 2 1/4, graphite, and weldment corrosion samples in the hot (in-pile) and cold (out-pile) sections. These samples will be machined, measured, and weighed prior to insertion in the loop as press-fit tubes. In addition, rod samples will be placed in the loop to determine effects of loop environment on the mechanical properties of Croloy 2 1/4.

After the loop operation is concluded, the loop components and contained samples will be removed and examined for corrosion, fission product absorption and/or adsorption, mass transfer, mechanical properties, and fuel penetration. Facilities for remotely removing, disassembling, packing, and shipping the component parts will be necessary.

1. GENERAL PROCEDURE FOR IN-PILE LOOP OPERATION

Following is a preliminary concept of the procedures necessary for securing accurate and valid data from in-pile forced circulation liquid-bismuth corrosion test loops. Slight modifications in pre-conditioning run-in times and sampling frequencies may be imposed as a result of out pile testing performed at the B&W Research Center in Alliance, Ohio.

After initial startup, loop procedures should require no interruption of reactor operation. Sampling will be accomplished remotely and sample analysis should be performed at the reactor site, if possible.

In the event of loop stoppage because of plugging or pump failures, a cooling medium will be circulated past the loop system to eliminate the necessity of reactor shutdown.

a. Startup

1) Preheat loop to 887 F, evacuate and out-gas

2) Melt bismuth in melt tank

3) Add bismuth to loop through filter

4) Circulate isothermally

5) Check pump performance

6) Measure flow velocity

b. Pre-Conditioning

1) Add 350 ppm Mg and circulate isothermally for 100 hours

2) Add 350 ppm Zr

3) Circulate isothermally for 100 hours

4) Sample to check concentration of additives

5) Add 1500 ppm U

6) Sample to check concentration of U and additives

c. Testing at Conditions

1) Apply thermal gradient by cooling cold section

2) Maintain temperature record, checking gradient maximum temperature every four hours.

3) Maintain constant flow metering

4) Sample every 48 hours for the first 250 hours and every 100 hours thereafter to check U, additive, corrosion product, and fission product concentrations

d. Discontinuance of test

1) Cease loop operation for any one of the following reasons:

(a) Completion of 5000 hour run

(b) Pump failure

(c) Rapid temperature rise as a result of plugging

(d) Reduction of flow as a result of plugging

2. DYNAMIC IN-PILE LOOPS (Radiation Loop #1 and #2)

Objectives:

a. To demonstrate the feasibility of the selected design parameters

b. To determine the effects of radiation on the corrosion of structural materials by U-Bi fuel solutions.

Conditions and Parameters: *

a. Material of construction: Croloy 2 1/4

b. Loop size: 3/4" (40) ips

c. Test fluid: U-Bi with additions of Zr and Mg

d. Temperatures: maximum 885 F, Δ T 135 F

e. Flow: eight to ten fps

f. Heat treatment: to be determined

g. Test duration: 5, 000 hours

* Conditions and parameters may be altered if out-pile test results indicate changes should be made.

3. DYNAMIC IN-PILE LOOP (Radiation Loop #3)

Objectives:

a. To obtain supporting data for Brookhaven Radiation Loop #1.

b. To demonstrate the feasibility of the higher temperature design parameters.

Conditions and Parameters: *

a. Material of construction: Croloy 2 1/4

b. Loop size: 3/4 inch (40) ips

c. Test fluid: U-Bi with additions of Mg and Zr

d. Temperatures: maximum 975 F, Δ T 225 F

e. Flow: eight to ten fps

f. Test duration: 5, 000 hours

* Conditions and parameters may be altered if out-pile test results indicate changes should be made.

SUMMARY OF

MATERIALS TESTING PROGRAM FOR LIQUID METAL FUEL REACTOR EXPERIMENT

THE BABCOCK & WILCOX COMPANY
Atomic Energy Division
June 1, 1957

AEC Contract AT(30-1)-1940
B&W Contract 600-0046

SPECIFICATIONS FOR URANIUM-BISMUTH FORCED CIRCULATION LOOP TESTS

REFERENCE CONDITIONS		ALTERNATE CONDITIONS
	MATERIALS	
Croloy 1¼		Croloy 1¼
Croloy 2¼		Croloy 2¼
Graphite		Graphite
885F	MAXIMUM TEMPERATURE	975F
135F	T hot - T cold (ΔT)	225F

TEST No.*	PRIMARY PURPOSE	TEST VARIABLES			TEST No.*
		HEAT TREATMENT	ADDITIVES (ppm)	VELOCITY (fps)	
1 2 3 / 10 11	Uninterrupted Demonstration Run	Annealed	Zirconium-350; Magnesium-350 Uranium-1500	8-10	6 7 / 14 15
4A 4B 4C / 12A 12B 12C	Corrosion Rate Determination	Annealed	Zirconium-350; Magnesium-350 Uranium-1500	8-10	8A 8B 8C / 16A 16B 16C
5A 5B / 13A 13B	Effect of Heat Treatment	Normalized and Tempered	Zirconium-350; Magnesium-350 Uranium-1500	8-10	9A 9B / 17A 17B
5C 5D / 13C 13D	Effect of Additives	Annealed	Th, Ca, Mg, Ti, Zr, U	8-10	9C 9D / 17C 17D
5E 5F / 13E 13F	Effect of Velocity	Annealed	Zirconium-350; Magnesium-350 Uranium-1500	4-5	9E 9F / 17E 17F

*Loop material for Test Nos. 1 through 5F and 6 through 9F is Croloy 2¼

*Loop material for Test Nos. 10 through 13F and 14 through 17F is Croloy 1¼

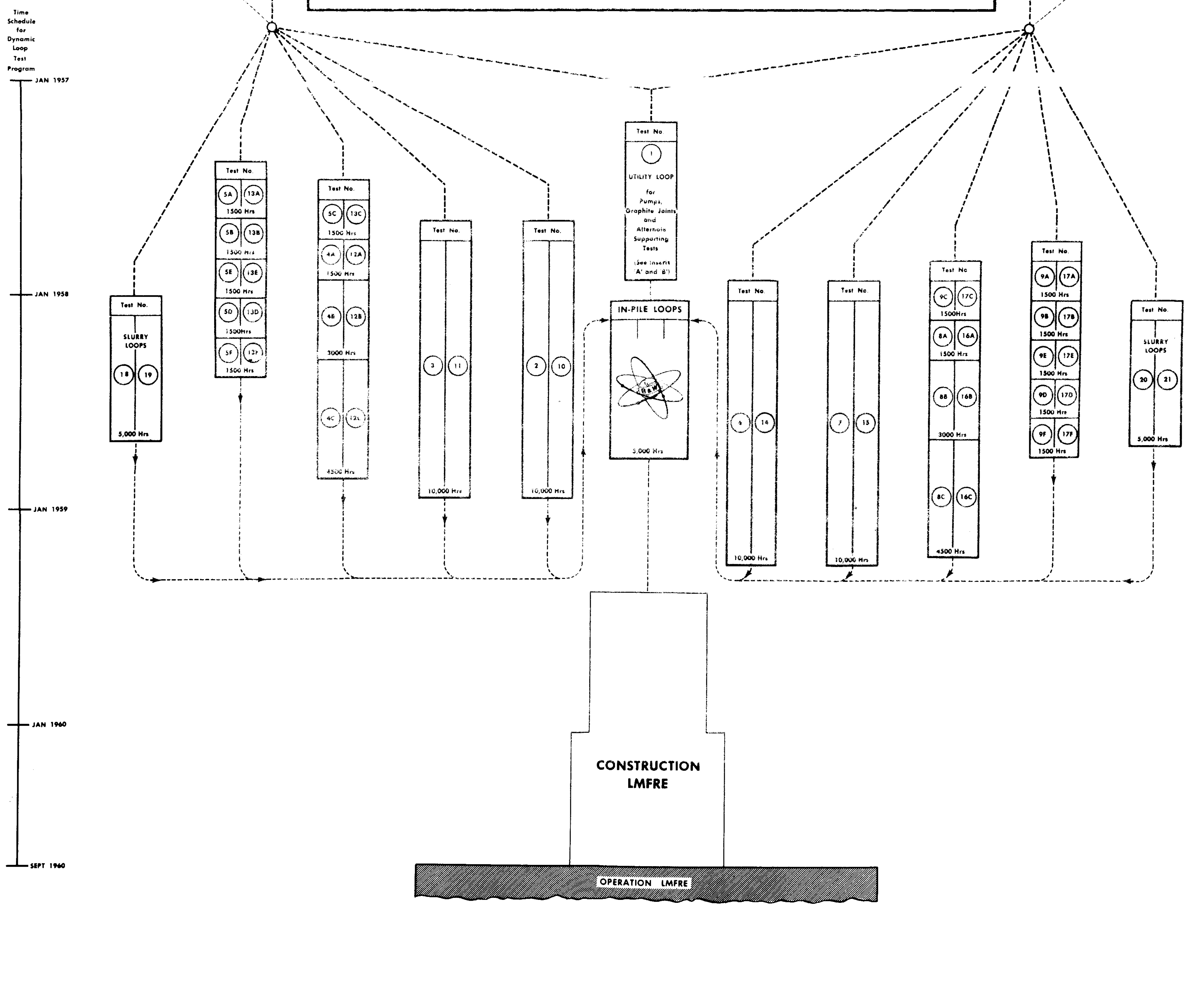
NOTE: Each loop will contain graphite, weldments, and loop material test specimens

SUPPORTING STATIC AND DYNAMIC TESTS

1. Effect of Heat Treatment on Corrosion and Mass Transfer
2. Effect of Additives on Corrosion
3. Effect of Conduit System Cleanliness on Corrosion
4. Effect of Fission and Corrosion Products on Corrosion and Mass Transfer
5. Investigation of Other Factors Influencing Mass Transfer
6. Investigation of ZrN Film Stability and Inhibitive Action

SUPPORTING STATIC AND DYNAMIC TESTS

7. Evaluation of Factors Influencing Fabrication Procedures
8. Investigation of Factors Influencing Wetting
9. Evaluation of Materials for Chemical Processing Equipment
10. Determination of Properties and Fabrication Limitations of Impervious Graphite
11. Investigation of Fuel Penetration and Outgassing of Graphite
12. Evaluation of Other Materials Suitable for use in the LMFRE



III. PROTOTYPE TESTING

III. PROTOTYPE TESTING

A. The primary objective of this program is to demonstrate the suitability and reliability of the various components which will constitute the Liquid Metal Fuel Reactor Experiment. The program includes investigation of graphite to metal seals, beryllium to beryllium to Croloy joints, various types of valves, and reactor fluid dynamics. As the design of the LMFRE proceeds, tests of other components probably will be necessary and will be added to this section of the R & D Program.

The reference construction material, i.e., Croloy 2 1/4, will be used in all tests except as specified to the contrary. The bismuth used in these tests will contain uranium and the proper inhibitors. In addition, construction materials exposed to the U-Bi will be pre-treated in accordance with established practice. Components will be tested at a maximum temperature of 977 F.

B. TESTS TO BE PERFORMED

1. GRAPHITE TO METAL SEALS (Test #1)

Objectives:

a. To simulate as nearly as possible the steady-state and transient conditions of expected reactor operation.

b. To test the effectiveness of the graphite to metal interface against bismuth flow with various bearing loads and at various fluid pressures.

c. To establish time rates of leakage.

d. To check the bismuth penetration into graphite.

Conditions:

a. Temperature - 977 F (max.) - 700 F (min.)

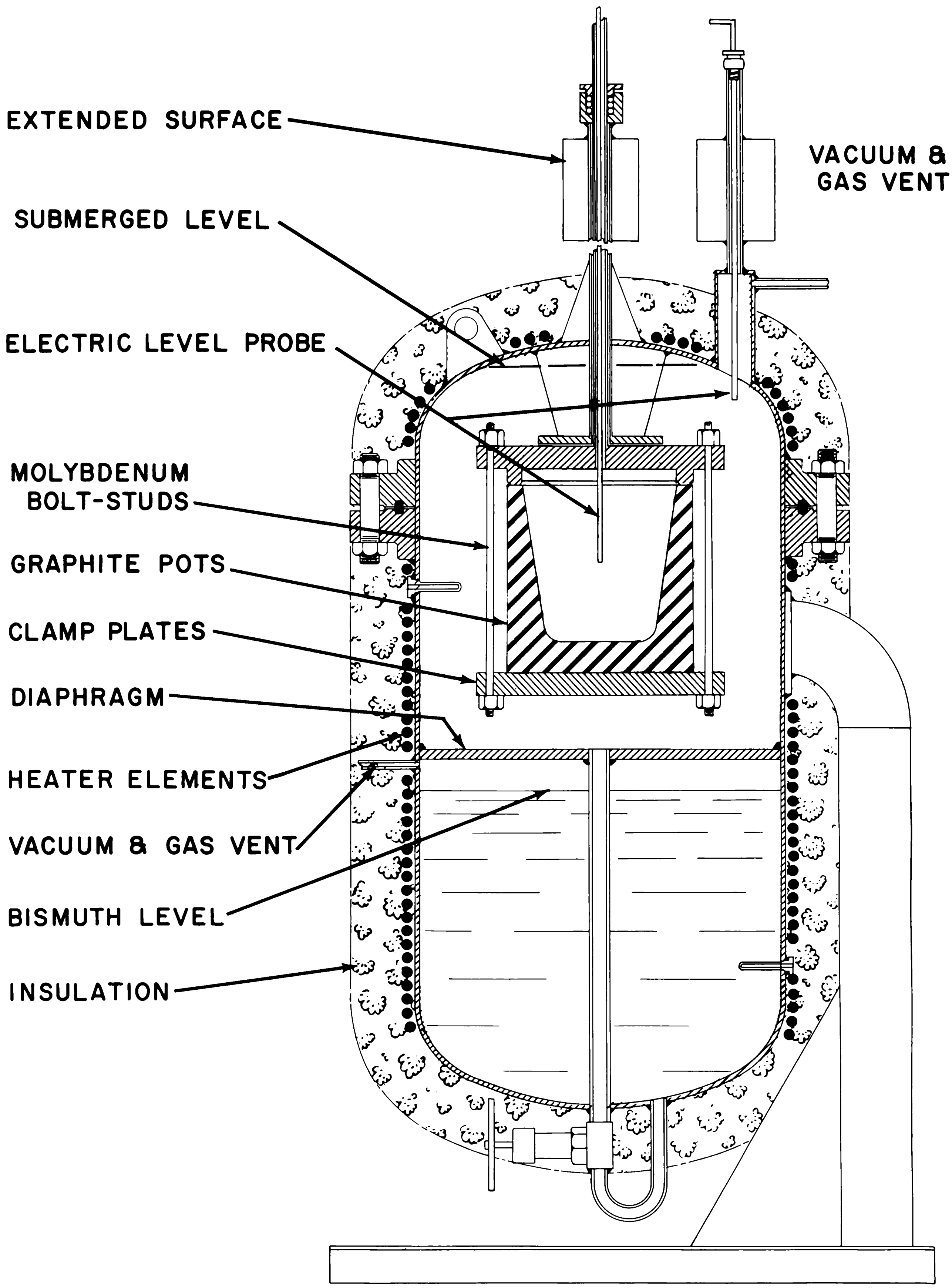
b. Pressure - 90 psi

c. Cycling between minimum and maximum temperatures. (approximately 200 cycles)

d. Approximately a quarter size model.

e. Initial requirements will be for four types of seal assembly. (probably two of each)

FIG. 4: CROLOY TO GRAPHITE SEAL TEST APPARATUS



2. REACTOR PORT THIMBLE JOINTS (Test #2)

Objectives:

a. To develop Croloy to beryllium joints that are 100 per cent leak tight.

b. To develop beryllium to beryllium joints which will be 100 per cent leak tight.

Conditions:

a. Temperature - 977 F (max.)

b. Pressure differential - 90 psi

c. Inside diameter of thimble - 4 inches

d. Length of test - 200 hrs.

The maximum length beryllium tube of four inches ID now available is four feet. It is necessary therefore to join sections of this tube in order to obtain the proper length. These joints may be screwed or flanged. It is felt that something in the nature of a pipe coupling may work.

It is possible that a two-section capsule joined with the joint to be tested will be a workable test sample. The capsule can be filled with liquid bismuth, pressurized and then cycled between 752 F and 977 F.

3. HYDRAULIC MOCK-UP OF CORE (Test #3)

Objective:

a. To investigate flow distribution, orificing and other reactor fluid dynamics.

It is possible that a plastic prototype model can be made utilizing a practical fluid such as water.

4. ABRASION TEST (Test #4)

Objective:

a. To test the abrasive effect of Croloy 2 1/4 and molybdenum on graphite.

b. To test the abrasive effect of Croloy 2 1/4 and molybdenum on each other.

Conditions:

a. 1000 to 2000 psi bearing pressure

b. Dry

c. In bismuth

d. After operation in bismuth

4. ABRASION TEST (Test #4)

Objective:

a. To test the abrasive effect of Croloy 2 1/4 and molybdenum on graphite.

b. To test the abrasive effect of Croloy 2 1/4 and molybdenum on each other.

Conditions:

a. 1000 to 2000 psi bearing pressure.

b. Dry

c. In bismuth

d. After operation in bismuth

5. DUMP VALVE (Test #5)

Objectives:

To determine dump valve dependability for the LMFRE.

Test should determine the following:

a. Reliability with successive dumps

b. Corrosion and erosion effects

c. Self welding

d. Leak tightness

e. Seal dependability

Conditions and Parameters:

1. The dump valves will be subjected to the following conditions:

		LMFRE	LMFR	TEST LOOP
a.	Valve size	2 1/2 in.	6 in.	2 1/2 in.
b.	Temperature range	750F-975F	750F-975F	750F-885F-975F
c.	Pressure across closed valve	70 psig	95 psig	70 psig
d.	Maximum dump velocity	22.5 fps	36 fps	22.5 fps-36 fps
e.	Average dump velocity	16.6 fps	22 fps	16.6 fps-22 fps
f.	Dump time	5 min.	5 min.	2 min.
g.	Quantity of UBi dumped	165 $ft.^3$	1400 $ft.^3$	60 $ft.^3$
h.	Head	16 ft.	24 ft.	
i.	No. of dumps	50	700	500

2. The valve chosen for this test should be such that self-welding possibility is minimized, and velocity and turbulence effects

held to a minimum. Specifically, the valve should be designed keeping a-e under above objectives in mind. To keep bismuth inventory low for this test, the valve should be quick opening with a maximum opening time of approximately two seconds.

3. Two, and possibly three series of tests will be necessary with temperatures at 750 F, 975 F and 885 F.

4. By varying either the U-Bi head or the overpressure, two theoretical initial velocities could be tested ----- 22.5 fps. and 36 fps.

6. LARGE CHECK VALVES (Test #6)

Two 12 inch check valves are required for pump isolation on the LMFRE. These valves will operate from 885 F to 975 F and will be closed with a U-Bi backpressure of approximately 100 psi. A leakage rate of several gpm is required to keep the idle line hot.

It may be possible to test this valve in the dump valve test loop if a pump of sufficient capacity can be obtained to give required velocities. The valve would be located in the supply tank fill line and would be required to hold up a 16 ft. head (70 psi) of U-Bi between the time the supply tank is filled and the dump valve opened. If it is possible to extrapolate data from a smaller size valve, e.g. four inches, to the required size, it would be desirable to test the smaller valve. If a four inch valve can be used with a velocity of 5 fps the flow time would be 2.3 minutes.

7. SMALL VALVES (Test #7)

A number of one inch isolation valves are required in the LMFRE. These will consist of both gate or globe type valves and check valves. Most of these valves normally will operate in the closed position, and be subjected to pressures of $\sim$ 150 psi, and temperatures as high as 975 F. Flow velocities through these valves should not exceed 5 fps in the "full open" position.

It is proposed to test these types of valves in the utility test loop.

Air actuators will be used on globe valves.

8. LARGE Bi PUMP TEST (Test #8)

Objective:

To determine large pump dependability and operating characteristics for a LMFRE.

The test should determine the following:

a. Cavitation data (required net position suction head as a function of flow and rpm)

b. Efficiencies at various loads.

c. Seal integrity.

d. Reliability of level control system.

e. Pre-heating characteristics.

f. Capacity(flow vs. head).

g. Electrical load vs. flow.

h. Minimum operating speed.

i. Responses to load changes.

j. Drainability.

Conditions and Parameters:

a.	Type of pump	Centrifugal
b.	Arrangement	Vertical
c.	Design pressure	300 psi
d.	Power requirements	440V 3 phase 60 cyc.
e.	Pipe size	12" sch. 30
f.	Design temperature	1050 F
g.	Type of pre-heating	Resistance wire
		Max. Min.
h.	Flow	2900 gpm (determine)
i.	Temperature	975F & 885F 750F
j.	Head	25' (108 psi)
h.	Variable suction pressure	14.7 psi to 100 psi

9. DEGASSER (Test #9)

Objectives:

a. To determine the most reliable, efficient, and practical type degasser for use in the LMFRE.

b. Measure iodine, zenon, and krypton removal rates as functions of temperature, pressure, flow, turbulance, sweeping gas flow rate, concentration, and degasser size.

c. To determine the most efficient and easily controlled method of varying gas removal rate for LMFRE physics experiments.

Conditions and Parameters:

a. Temperature range	750F to 975F
b. Pressure range	13 to 40 psia
c. Bi flow rate	2900 gpm
d. Sweeping gas flow rates	1 to 100 ft^3/hr.
e. Zenon conc. range (fuel)	1.5×10^{-3} ppm (based on 2% removal)
f. Zenon removal rates reqd.	2%

IV. INSTRUMENTATION

A. REACTOR AND RADIATION INSTRUMENTATION AND CONTROL DEVELOPMENT

The instrumentation and control items which will require development necessary for efficient operation of the LMFRE include:

1. High-temperature neutron detectors
2. A continuous uranium monitoring system
3. High-temperature radiation instrumentation
4. A reactor seal monitor
5. Revised electronic circuits

The development of the above items is necessary for efficient control and adequate indication of the essential parameters for the experiment. The high temperature equipment will allow operation without the use of auxiliary cooling equipment which would be a source of difficult and costly maintenance. In the absence of any cooling devices, the integrity of the system is dependent only upon its primary components which are designed to operate in the required ambient temperatures.

The continuous uranium monitoring system will measure and control the uranium concentration in the reactor and/or the oxidation potential of the salts used in the chemical processing plant, while the seal monitor will indicate any leakage which may occur between the reactor and reflector streams.

The revised electronic circuits will be used in conjunction with the new high-temperature instrumentation. These circuits will increase the reliability and simplify the maintenance of the reactor and control equipment.

The development program will be accomplished by submitting preliminary specifications for proposals to qualified manufacturers. The chosen manufacturer(s) will then enter into a sub-contract to perform the required work. The program in general is broken down into the following phases.

1. Study phase, which will determine the most likely materials and methods to be used.
2. Preliminary design phase, to establish geometries.
3. Component testing phase, to establish material parameters at operating conditions.
4. Final design phase, in which a prototype will be designed and fabricated.
5. Testing phase, to determine if the prototype operates satisfactorily.
6. Evaluation phase, to attempt to re-evaluate the design and improve prototype if necessary.

The testing will demonstrate the material or component design and may include transient response, temperature, sensitivity, linearity, accuracy, fatigue, or compatability testing.

1. HIGH-TEMPERATURE NEUTRON DETECTORS (Test #1)

If present multi-detector schemes are to be employed for the

LMFRE, at least two types of sensors will have to be developed.

a. The start-up detector, which will operate from source power to approximately 10^5 nv, will be capable of operating continuously in an ambient of 1000 F and will have a minimum lifetime of 1×10^{12} counts. The detector will operate in gamma intensities of 10^5 R/hr without appreciable decrease in sensitivity. It will have a useful range of at least five decades with a minimum sensitivity of 0.5 c/nv. The material used for the detector housing will have a low-neutron capture cross-section and will be compatible with the insulators under thermal expansion. Sufficient cable (approximately 20 feet) will be furnished as an integral part of the detector to eliminate any screw connection at the detector. The negative output pulse from the detector will be a minimum of 100 micro-volts with a rise time equal to or less than 0.2 micro-seconds. Either a high-neutron capture cross-sectional coating or gas filling may be employed although no restrictions are placed on the detector as to type. It is expected that either a fission chamber or proportional counter can be developed to meet the specifications.

b. The intermediate and power range detector(s) will cover the neutron range from 5×10^3 to 5×10^{10} thermal neutrons/cm^2/sec and will operate continuously in ambients up to 1000 F. The detector(s) should have a minimum lifetime of one year at the maximum flux and operating temperature. Approximately 25 feet of cable will be supplied with the detector to carry necessary voltage and signals. The cable will be an integral part of the detector in order to eliminate screw connections. The housing material used for construction will have a low-neutron capture cross-section and will not cause excessive distortion of critical dimensions when heated to the operating temperatures.

If an ionization chamber is employed, it will meet the following additional requirements. The chamber will have a minimum sensitivity of 5×10^{14} a/nv with an uncompensated gamma current not to exceed 3×10^{11} a/R/hr. A minimum lifetime of 5×10^{18} nvt will be expected. If gamma compensation is necessary, the chamber will be compensated electrically and the gamma current reduced at least two decades. The chamber may be operated either compensated or uncompensated, but when operated uncompensated, only the top four decades need be covered.

2. CONTINUOUS URANIUM MONITORING SYSTEM (Test #2)

This system will measure and control the uranium concentration in the reactor and/or oxidation potential of the salts used in the chemical processing plant. The monitoring system will be capable of indicating uranium concentrations from 10 to 2, 000 ppm. The instrument can be used as one of the control parameters for reactor power and as an indication of uranium concentration and/or absorption. The operating temperature of the liquid to be monitored will be between 750 F and 1200 F and the instrument will have an accuracy of ± 2% with a minimum response time. The uranium monitored will be in either the reactor fuel stream, i.e., enriched U^{235} and bismuth plus 500 ppm zirconium and magnesium solution, or in an alkali and/or alkali earth halide solution.

3. HIGH-TEMPERATURE RADIATION INSTRUMENTATION (Test #3)

This type of instrumentation will be used as a control and indication or radiation levels throughout the plant where standard instrumentation cannot be used; the instrumentation will be capable of withstanding temperatures up to 1000 F and will maintain sensitivities of ± 2% over the lifetime of the instruments.

Levels from 10 mr/hr to approximately 10^5 R/hr will be measured and the indications used either for automatic or manual control of various processes in the chemical process plant, accessibility to reactor or system components, wastes and plant effluents.

4. REACTOR SEAL MONITORING SYSTEM (Test #4)

This system will monitor leakage across the seals between the reactor coolant and reflector streams. It will be capable of indicating both trends and percentage leakage, thus proving the design of the seals. The monitoring system will be useful in determining if blanket slurries can be used in the reflector without leakage across the installed seals. The instrument will be capable of operating with liquid streams of temperatures from 750 F to 1000 F.

5. REVISED ELECTRONIC CIRCUITS (Test #5)

New electronic circuits will have to be engineered and designed in order to be compatible with the high-temperature instrumentation which is developed. These electronic circuits will employ the newest and most reliable components. At this time, it is impossible to determine the exact content of the electronic circuits to be developed, but where possible, magnetic amplifiers or transistors will be used if later simplicity or reliability can be obtained. It also may be necessary to design and build electronic equipment which will be compatible with temperatures up to approximately 1000 F. These high-temperature electronic circuits then could be placed in the same ambients as detectors, thus eliminating any need for pre-amplifiers which may be situated at great distances from the sensing elements.

6. TEMPERATURE MEASURING DEVICES (Test #6)

Resistance elements and thermocouples are the most promising means of measuring temperature. The predominent problem associated with both instruments is obtaining a sheath material compatible with the system fluid and the temperature indicator design.

The nature of the reactor may necessitate the provision of rapid response temperature sensing instruments. These instruments should have a response time of 0.1 - 0.5 sec. in order to follow system transients.

Thermocouples will be tested first under static conditions to determine their operating characteristics and then under dynamic conditions to determine life times.

Objectives:

a. To select and obtain operating characteristics of thermocouples suitable for bismuth service under LMFRE requirements.

b. Tests should determine the following:

1. Reliability
2. Response time
3. Accuracy (accuracy variation with time)
4. Sensitivity
5. Replacement considerations (remote maintenance)
6. Leak tightness

Conditions and Parameters:

	Rapid Response	Standard Response
a. Overall range	600-1200 F	600-1200 F
b. Operating range	750-975 F	750-975 F
c. Response (62.3% of change)	0.1-0.5 sec.	2 sec.
d. Design Pressure	200 psi	None
e. Testing Time	2-6 months	1-2 months
f. Accuracy	Deviations from true readings to be maintained not greater than ± 1% over the operating range.	

Instrument Types to be Tested:

a. Rapid Response Thermocouples.

1. Chromel-alumel (swaged) magnesia insulated thermocouple. Sheath and weld material Croloy 2 1/4. Hot junction welded to sheath.

2. Iron-Constantan - same as above.

3. Resistance temperature detector. Plantinum resistant element silver soldered to external leads. Platinum element incased in Croloy 2 1/4 and seal welded to base.

b. Standard Response Thermocouples

1. Chromel-alumel (swaged) magnesia insulated surface thermocouples. Sheath and weld material Croloy 2 1/4. Hot junction welded directly to pipe.

2. Iron-Constantan - same as above.

7. PRESSURE MEASURING DEVICES (Test #7)

A number of pressure indicators with various ranges will be required for bismuth service. They may be used as liquid-level indicators and flow-meter components as well as standard system pressure indicators.

These instruments may require a special test loop to obtain the necessary operating conditions for instrument screening tests and instrument operating data. Long term operational characteristics may be obtained by installing gages in material or utility test loops.

Objectives:

a. Selecting and calibrating pressure-indicating instruments suitable for bismuth service.

b. Tests should determine the following:

1. Reliability
2. Response time
3. Accuracy
4. Effects of temperature
5. Leak tightness
6. Sensitivity

Conditions and Parameters:

a. Overall range: 0-200 psi (must take short duration, rapid, pressure rise to 100 psi above operating pressure)

b. Operating range: atmo-150 psi

c. Response: less than 1 sec.

d. Accuracy: deviations from true reading to be maintained not greater than ± 1% over the operating range.

e. Testing time: 6 months (estimated)

f. Design temperature: 1050 F

Instrument Types To Be Tested: .

a. An inductance type pressure gage consisting of a sealing bellows in contact with the bismuth and connected by a rod to a measuring springbellows combination. Attached to the measuring combination is a compensated differential transformer.

b. A pressure transmittor consisting of a diaphragm separating the bismuth from a capillary tube containing NaK. The capillary tube extends to a bourdon tube.

8. LIQUID LEVEL MEASURING DEVICES (Test #8)

A number of liquid level indicators with various ranges will be required for bismuth service. These instruments will be tested in the dump valve test loop.

Objectives:

a. Selecting and calibrating a level indicator(s) suitable for bismuth service.

b. Test should determine the following:

1. Reliability
2. Response Time
3. Accuracy
4. Sensitivity
5. Effects of temperature and pressure
6. Leak tightness
7. Replacement considerations (remote maintenance)

Conditions and Parameters:

	Overall level variations	Operating level variations
a. Range:		
Indicator No. 1	4 feet	Unknown
Indicator No. 2	1 feet	3 to 6 inches

b. Response time: less than 1 sec.

c. Accuracy: Deviations from true reading to be maintained not greater than ± 5 per cent throughout the overall range

d. Design Pressure: 200 psi

e. Design Temperature: 1050 F

f. Testing time: 6-12 months (estimated)

Instrument Types To Be Tested:

a. A "Torque Tube" level indicator consisting of a float to sense level action and action supplying a movement to a control system through a torque tube.

b. Resistance level probe

c. Fixed contact level probes

d. Buoyant - differential transformer type consisting of a buoyant type sensing element connected mechanically to a differential transformer

9. FLOW MEASURING DEVICES (Test #9)

Flow measurements will be required in the main bismuth stream and in auxiliary systems handling bismuth.

The smaller size flow meters (3/4" to 2") will be tested in the utility test loop. Screening tests for the larger meters will be performed in the dump valve loop (2 1/2"). Data obtained from tests in the utility loop will be used to select meters for testing in the dump loop. Large meters (12 inch), selected from the screening tests, will be tested in the Large Pump Test Loop.

Objectives:

a. To select and obtain calibration data for various size flow meters suitable for bismuth service under LMFRE conditions

b. Tests should determine the following:

1. Reliability
2. Response time
3. Accuracy
4. Sensitivity
5. Effects of temperature and pressure
6. Replacement considerations (remote maintenance)
7. Leak tightness

Conditions and Parameters:

a. Meter size range:	3/4 inch to 12 inch
b. Overall range:	0-3000 gpm
c. Response:	less than 1 sec.
d. Accuracy:	deviations from true reading to be maintained not greater than ± 1 per cent over the operating range.
e. Design temperature:	1050 F
f. Design pressure:	200 psia
g. Testing time:	1 year (estimated)

Instrument Types To Be Tested:

a. A flow sensing element consisting of a friction-free turbine type rotor with a built-in permanent magnet which induces an alternating current in a pick-up coil mounted on the exterior of the sensing element housing.

b. Orifice flowmeters - to be used in conjunction with a pressure gage

c. Venturi flowmeters

d. Calibrated elbow flow meter

e. Magnetic flowmeter

f. Electro magnetic flowmeter

g. Ultrasonic type flowmeter

V. CHEMISTRY

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A. INTRODUCTION

Numerous chemical problems have been anticipated in preparing the LMFRE development program. These generally fall into the fields of (1) fuel processing, including both the fuel and fertile material, (2) analytical chemistry, involving control analyses of the fuel and heat transfer fluids, and (3) reactor chemistry, including corrosion phenomena, radiation chemistry and chemical hazards. Other chemical problems which may arise during the course of the work will probably fall into these same fields. In the following treatment the research and development efforts required for each of these fields have been presented and discussed in some detail.

B. CHEMICAL PROCESSING

An ideal full scale LMFR power station will probably utilize a breeder reactor operating with a uranium-bismuth solution as a fuel and a suspension of thorium bismuthide in liquid bismuth as a breeder blanket fluid. Accordingly, a demonstration of processing methods for both the fuel and fertile material are desirable objectives for the LMFRE. The development program should be sufficiently broad that full scale equipment for a large power station can be designed with a minimum of extrapolation.

At the present time, there is no apparent "best" method of processing. In fact, no existing system can be used with confidence in conjunction with the experiment. For this reason several methods of processing which may lead to a feasible chemical plant design for the experiment should be considered.

The following processing operations will be studied in the research and development program:

1) Make-up of fuel. Provision will be made for the controlled addition of uranium and other constituents of the fuel.

2) Clean-up of bismuth. Rapid processing of the bismuth will be provided for removal of natural uranium added to condition the primary loop. The process also will be used to lower the uranium concentration, to recover bismuth and to recover U235 at the conclusion of the reactor experiment.

3) Removal of volatile fission products. Degassing of the primary fluid and storage facilities for the volatile waste will be provided.

4) Extraction of fission products which form halides more stable than bismuth halides. Three alternative processes will be studied:

a. Fluoride volatility processing, utilizing the volatility of uranium hexafluoride to separate uranium from fission products.

b. Fused chloride extraction, incorporating selective transfer of fission products and uranium between bismuth and salt phases.

c. Modified fused chloride extraction (electrolytic system), utilizing controlled voltages to achieve selective transfer of fission products or uranium between bismuth and fused salt.

5) Removal of the fission products not susceptible to removal by chemical reaction. Metal-metal extraction, cold-trapping, distillation and intermetallic slagging are being considered.

6) Transfer of U-233 from solid $ThBi_2$ to liquid bismuth. Further processing of the resulting solution into some form that may be charged to the core also is required.

1. FUEL MAKE-UP SYSTEM

Provisions must be made in LMFRE for the periodic or continuous make-up of uranium and other compounds of the fuel. Two general methods currently are being considered for this purpose:

a) Direct dissolution in flowing bismuth.

b) Transfer of the additives from a fused salt bath to the fuel.

The former method probably can be made operable on a shorter time scale than the latter, but would not be as adaptable to automatic control. In the latter scheme molten bismuth is contacted by a fused salt containing the additives. The transfer of material from the salt phase to the bismuth can be brought about by reduction, either by electrolysis or by a metal such as calcium dissolved in the bismuth.

Such a system could be integrated easily into the rest of the chemical processing plant. Furthermore it is possible that a voltage measurement between the salt and the fuel can be interpreted as concentrations of additives.

A research and development program is required to demonstrate fuel make-up methods, however, the bulk of the work can probably be done on a bench scale.

2. RAPID BISMUTH CLEAN-UP

A processing scheme for removing uranium from bismuth must be provided at the time of the LMFRE start-up. The equipment will remove natural uranium added during the pre-conditioning and will provide a means for lowering uranium concentrations during reactor operation.

Several slagging operations appear suitable. Caustic slagging with NaOH and $NaNO_3$ as used in bismuth refining can probably be adapted for this purpose. Fused salt extraction with metal chlorides is already well underway in AEC laboratories. Fluoride slagging, the first step in a volatility process, appears to be feasible for early development.

Chloride and fluoride processes are favored over caustic slagging because they offer promise as initial steps in pyro-processes whereas caustic slagging appears to lead only to aqueous processing. In view of the over-all LMFR program, it seems advisable to concentrate the development of continuous pyro-processing with its attendant lower costs.

The development of any of these processes requires a study of reaction kinetics, removal efficiency, contactor dynamics, and corrosion. The operation of a pilot scale demonstration will be required before a process is used on the LMFRE.

3. VOLATILE FISSION PRODUCT REMOVAL

The system for processing volatile fission products has two major sections, a degasser and a unit to recover the gases. The entire primary loop flow will pass through the degasser where the volatile materials will be removed. The gases will be diverted to adsorption beds.

A research program is necessary to determine the volatility of the components in the fuel stream. The small amount of available information indicates that the fission gases, xenon and krypton, are extremely insoluble in bismuth and that their removal may be accomplished readily from bismuth-gas interfaces. Since diffusion of the gases in bismuth is slow, the efficiency of the degasser will depend primarily on the available surface area and the turbulence of the bismuth; accordingly, the degasser will be designed to maximize both these quantities.

The degasser design will be developed in the research program. The initial work will be a study of the dispersion of gases and liquid metals. The first tests can be run with mercury and air in glass apparatus. As the design evolves, pilot scale apparatus using bismuth and helium will be tested. It may be possible to remove dissolved natural xenon and krypton from bismuth by some mass transfer tests. Small degassers will be incorporated in the in-pile corrosion test loops and these data will be used in the degasser design.

At this time it is agreed generally that the bulk of the degassing mass-transfer studies will be performed during the Reactor Experiment. Consideration is being given to operating a small degasser in parallel with the full-stream degasser to present a more flexible experimental tool.

The waste handling or off-gas system will receive the volatile fission products in a stream of helium from the degasser. The metals will be condensed from the stream and the fission gases will be adsorbed on activated charcoal. Helium stripped of the fission gases will be recirculated to the degasser.

The metals, principally bismuth and polonium, which are condensed in the cold trap will be returned to the fuel stream. The fission gases will be stored on the charcoal beds until all of the xenon and krypton, except the longer lived Xe^{131m}, Xe^{133m}, Xe^{133}, and Kr^{85}, have decayed. After they are removed from the beds, the gases will be stored in tanks until all radioactive species gases, except Kr^{85}, have decayed. At this time, the gases will be vented to the atmosphere.

Considerable study will be required to determine the performance of activated charcoal in adsorbing xenon and krypton from helium. The effects of radiation and metallic decay products on bed capacity will be examined in the small off-gas system which will be incorporated with the degassers in the in-pile corrosion loops.

4. EXTRACTION OF REACTIVE FISSION PRODUCTS

Fission products which form halides more stable than bismuth halides can be extracted from bismuth by slagging techniques. Three variations of this scheme are being considered for the LMFRE fuel: 1) Fluoride volatility processing in which uranium is recovered from a fluoride slag by volatilization of uranium hexafluoride; 2) Salt extraction in which selective extraction is accomplished by control of oxidation potentials permitting uranium recovery; 3) Electrolytic processing, a modification of the salt extraction process.

Fluoride Volatility Processing - The volatility process may be divided into four steps; primary fluorination, secondary fluorination, uranium fluoride reduction, and fuel make-up. In the primary fluorination, core fluid is treated to convert uranium and fission products to insoluble fluorides which are extracted by a fused-fluoride phase. The fluoride salt phase is further treated in the second fluorination step by gaseous F_2 which converts UF_4 to UF_6. The volatile UF_6 is recovered by condensation and the decontaminated UF_6 is reduced to the UF_4 with hydrogen. The UF_4 is dissolved in a fused salt phase and the uranium is transferred into bismuth fuel by either chemical or electrolytic reduction.

The major new research required is the development of fluorination in the presence of bismuth and the reduction of UF_4 to metallic uranium. Fluoride volatility techniques have received considerable study by the AEC and their successful application to LMFR fuels seems very promising. An integrated process would be demonstrated on a pilot plant scale prior to testing on the LMFRE.

Fused Chloride Extraction - The chloride extraction process achieves separation of components in the U-Bi fuel by utilizing differences in their reactivities. Metallic fission products which form chlorides more stable than bismuth chloride can be oxidized at the bismuth-salt interface to chlorides which then dissolve in the salt phase. Non-metallic fission products, e.g., iodine, bromine, telurium and selenium also are expected to transfer into the salt, but in their reduced state. Theoretically, under careful control of oxidation potentials, the fission products more reactive than uranium (Rb, Cs, Ba, Sr, Y, La, and rare earths) can be extracted from the bismuth with minimum uranium extraction.

Selective reduction of uranium and fission products also can transfer components in the other direction, that is, from salt to bismuth. Thus it is possible to transfer groups of solutes back and forth between metallic bismuth and fused salts almost at will. A series of multi-stage oxidation and reduction units can be incorporated to obtain the desired fuel processing results.

A research program must develop methods for sensing and controlling the oxidation potentials which are so critical to the success of this scheme. The effectiveness of various oxidizing and reducing agents ($BiCl_3$, $MgCl_2$, Mg. Ca, and others) will be studied.

A definite need exists for more fundamental data such as equilibrium distributions and activity coefficients for solutes in bismuth and fused salts. These data are currently being obtained at Brookhaven National Laboratory and the program is expected to continue at an accelerated rate.

Development is required for liquid-metal fused-salt contactors. The design of a liquid-metal fused-salt contactor is not a straightforward operation, primarily because little is known about the fluid dynamics and diffusion rates. A study of fluid dynamics and reaction and diffusion kinetics is indicated. Although some kinetic data will be determined in bench scale tests, these investigations will require loop scale operations. Before salt extraction is incorporated with the LMFRE, a process pilot plant should be operated.

Fused Salt Electrolysis - Electrolytic techniques have several attractive applications to an LMFR. The broadest application is a modified fused salt extraction process in which transfer of material between a fused salt layer and the fuel is controlled electrically. The system, which is readily adapted to remote operation, can thus be used for fuel make-up and permits adding uranium to the reactor as UF_4 which is considerably cheaper than uranium metal.

Electrolysis holds sufficient promise for the LMFRE to merit a research and development program investigating the possible applications. The preliminary work would be an investigation of the transfer of uranium between fused-salt and liquid-bismuth phases. This work would be followed by a study of the behavior of the fission products.

5. REMOVAL OF NOBLE FISSION PRODUCTS

In order to achieve complete bismuth clean-up and to minimize neutron poisoning in a long-lived LMFR, solutes which are less reactive than bismuth must be removed from solution. Although the isotopes which are generally considered the worst neutron poisons can be removed by degassing or chemical reactions, these noble fission products also will present a significant poisoning effect after prolonged reactor operation. Since the noble materials cannot be removed by chemical reaction, physical separations must be considered. Metal-distillation, cold-trapping, metal-metal extraction and intermetallic slagging will be studied as potentially applicable processes.

6. THORIUM-BISMUTH SLURRY PROCESSING

Thorium-bismuth slurry loops will be inserted in the LMFRE irradiation holes in order to study blanket processing. Many of the blanket processing steps will be similar to the fuel processing steps, but the one major difference results from the presence of solid particles in the blanket.

Bred material which forms inside thorium-bismuthide particles must be transferred to the liquid phase to permit uranium recovery. The transfer will be accomplished by solid diffusion or by fusion of the particles. Although diffusion appears too slow for practical processing, temperature cycling which results from heat generation in the slurry may greatly enhance the transfer by causing partial solution and regrowth of the particles. In the event that acceptable transfer rates can be obtained only by particle destruction, that is, by diluting with bismuth and increasing the temperature, reconstitution of the slurry may present a major problem. In any event, an investigation is required to determine the conditions which produce a slurry with a suitable particle shape and size distribution.

The research program will emphasize the reconstitution of slurries. Two techniques, electrolytic deposition of thorium in bismuth and rapid cooling of saturated solutions of thorium in bismuth, will be studied. Bench scale and loop scale apparatus will be developed.

The chemistry of processing the bismuth solution also requires study to examine the effects of protactinium and thorium on the schemes proposed for fuel processing.

C. ANALYTICAL CHEMISTRY

The successful operation of the Reactor Experiment as well as adequate interpretation of experimental results is completely dependent on chemical analyses. Fuel composition is a particularly important factor in the physics and metallurgy of the reactor plant. Nearly all of the analyses must be performed on highly radioactive samples.

The research program will supplement the existing analytical procedures for U, Zr, Mg, Po, fission products, and corrosion products in bismuth. Chemical methods to assist in interpreting system failures will be developed. Although the bulk of the analytical procedures required for the chemical plant will be developed for the pilot plant research, some supplementary techniques will require study.

D. REACTOR CHEMISTRY

The bismuth fuel solution containing additives and fission products in direct contact with the graphite in the core poses the possibility of many chemical reactions. The reactor system will be pre-treated with bismuth solution containing magnesium and zirconium and, later, natural uranium. Magnesium deoxidizes the system while zirconium conditions the container and graphite surfaces.

It has been demonstrated that, in the absence of radiation, uranium carbide does not form in measurable amounts at the design temperature conditions of our system, but zirconium carbide does form on the graphite surface. It is not known which of the fission products, if any, may react with this ZrC. Fission products will be in competition with the zirconium for exposed graphite surface. Free energy data for the formation of fission product carbides must be developed. The physical properties of carbide films must be studied, and in addition carbide formation rates from bismuth solutions require study. If high cross-sectional fission products concentrate on the graphite surfaces in spite of the presence of zirconium, the useful life of any one graphite core would be limited. The nature of the carbides which form must be investigated.

Radiation effects on the formation of carbides are unknown. Radiation may affect the nature of the product as well as the rates of reaction. Studies will determine whether reactions might become progressive rather than be inhibited by the initial carbide layers.

The effects of sodium leakage into the fuel must be considered. The reaction of sodium in bismuth on graphite and the effect of sodium on the solubilities of uranium, additives, and fission products must also be examined.

Based on test loop experience, it is anticipated that uranium losses will occur during initial start-up of the reactor system. Although the uranium used during the initial clean-up step will probably be U^{238}, after U^{235} is added to the bismuth isotopic interchange will take place. It is of interest to know the amount and nature of uranium which will be lost during initial conditioning and the rate and extent of isotopic interchange. The amount and character of uranium which is lost will be studied on the corrosion loop tests. Isotopic interchange rates will be determined in bench scale tests with uranium-bismuth solutions exposed to precipitated uranium.

The behavior of polonium in the reactor primary system and in the auxiliary processing system is largely unknown. Polonium is not expected to influence either the reactor physics or the corrosion problems encountered in the LMFRE. Because of its severe ingestion hazard and the (alpha, n) reaction which it creates in light isotopes, polonium must be considered as a major reactor hazard. Its fate in the various systems must therefore be determined as accurately as possible during the research and development work.

VI. REMOTE MAINTENANCE

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Preliminary development work will be necessary for a complete remote handling and maintenance program. The specific areas in which development is needed are not well defined at this time, but some of the work can be outlined in general.

The remote welding program will require R&D in the following areas:

1. The effects of residual bismuth entering the weld area during the welding operation.
2. The quality of induction or resistance push-up welded joints under the specific conditions of use.
3. The effects of the fuel stream on different remotely welded joints.
4. Other unknowns pertaining to remote welding will be explored.

Remote viewing for control of the remote welder and weld inspection will be explored. Closed circuit TV is the most promising approach at this time.

Associated information necessary for sound mechanical designs will be required as the various designs progress.

It is proposed that this work be performed by a qualified vendor under a sub-contract with preliminary development and possible back-up testing at the B&W Research Center.

VII. CRITICAL EXPERIMENT

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Proposed Experiments

In the course of preliminary design studies it has become apparent that the LMFRE should operate at a high fission density within the limitations of total power output. In order to maximize fission density, the initial U^{235} concentration in the fuel must be set as high as possible. For the reference design the initial U^{235} concentration has been set at about 1150 ppm., and at the minimum possible fuel temperature the uranium solubility is 1700 ppm $\pm$ 10%. After allowance is made for higher isotope buildup (220 ppm) and fission product poisoning (100-200 ppm) the margin for error in predicting the critical concentration is reduced to about 10-20%, depending on what assumptions are made about fuel processing efficiencies.

The B&W Physics and Mathematics Department has recently completed a survey of experimental data available on enriched uranium-graphite critical experiments. Unfortunately, only two separate experiments are available for analysis, and these experiments give contradictory results. Furthermore, no experiments have been run on small, highly reflected systems, such as are of interest to the LMFRE. On the basis of the limited amount of data available today, there is no adequate check on the methods of analysis that will be employed to calculate the critical concentration of the LMFRE. It is entirely possible that the error in predicting critical concentration could far exceed the 10-20% margin of safety available in the reference design.

It is proposed that a number of critical assemblies of enriched uranium graphite, and bismuth be constructed. The first experiments will be designed to meet the reference design parameters as closely as possible, e.g., simulate cylindrical geometry and maintain V(Bi)/V(C) - 0.5. In these experiments the critical size will be determined for several uranium concentrations and reflector thicknesses. If time permits, experiments will be performed on other V(Bi)/V(C) ratios, within the limits of available materials.

The critical assemblies will be built of blocks of bismuth and graphite alternated with thin strips of U or U-Al foil. The dimensions of the blocks will be carefully selected so as to permit a number of assemblies to be constructed with a minimum of different shapes. In order to conform with standard safe practices, the assemblies will be built in halves, each half provided with safety rods, and provision made for bringing them together remotely.